![GWTT logo] **GWTT**
Ground/Water Treatment & Technology, LLC

627 MT. HOPE ROAD
WHARTON, NEW JERSEY 07885
TEL: (800) 770-0901
(973) 983-0901
FAX: (973) 983-0903

January 12, 2016

DRAFT

Environmental Restoration, LLC.
1666 Fabick Drive
Fenton, MO 63062

Attention: Todd Conley

Reference: ERRS Region 2 WL2-26
Treatability Study
RFQ # WL2-26-04MB

Dear Todd;

The following is our report detailing the findings of a treatability study using different media for the reduction of lead in water discharging from the Wurtsboro Mine in Wurtsboro, NY.

Treatability Study Methodology

Approximately 40 gallons of water was collected from the discharge of the mine on Wednesday December 16, 2015. The water was transported back to our laboratory in Wharton, NJ where it was placed into a larger container and sealed at the top except for a small hole for the pump suction tubing.

Three different media were tested by placing the media in a clear PVC tube. A dutch weave screen was placed in the bottom of the tube to keep the media within the tube. A needle valve was placed in the bottom to regulate the effluent flow rate.

Chemical metering pumps were used to convey the water to the top of the tube. The flow rate into to the tube was matched to the flow rate out and the media was kept saturated during the entire test with the operating water level within the tube several inches above the media at all times. The water was collected in a 1,000 ml glass beaker and the total volume that passed through the media recorded as samples were collected.

A 1" diameter tube was used for the #16/60 limestone and Activated Alumina while a 2" diameter pipe was used for the 3/8" limestone (to reduce side wall effects).

Activated Alumina was used as one of the test media. This media was used based upon published reports that this media was able to reduce lead in water. The Activated Alumina was sourced from SORBEAD India.

ACTIVATED ALUMINA

Fine grained limestone was used as another test media. This media was used based upon published reports of its ability to reduce lead in water. This media has the general characteristics of 100 percent passing the #16 (1.19 mm) sieve while 100% is retained on the # 60 (0.25) sieve. This media was sourced from the Carver Stone in Schoharie NY located approximately 100 miles from Wurtsboro, NY

#16 / #60 LIMESTONE

3/8" limestone was used as the third media tested. This media was sourced from the Tilcon Rock Quarry in Oxford, NJ located approximately 70 miles from Wurtsboro. This media was tested because the concern over the physical size and low permeability of the 16/60 media.

3/8" LIMESTONE

Treatability Test Results

The untested water had a pH of 5.77 and a total lead concentration of approximately 500 ppb. The dissolved component was 475 ppb indicating that the majority of the lead is dissolved in the water.

The results of the treatability study are presented in the chart below and are summarized as follows;

1. The **Activated Alumina** was able to reduce the lead concentration in the water from 536 ppb to as low as 9.18 ppb; however, the reduction efficiency was reduced as the testing progressed so that after 500 bed volumes the lead concentration was 94.58 ppb; significantly above the discharge criteria for lead of 3 ppb. Given these test results the used of Activated Alumina is not recommended for this site.

2. The **Fine Grained Limestone** was able to reduce the lead concentration to approximately 0.5 ppb throughout the duration of the test with one anomaly. The use of this media meets the discharge criteria of 3 ppb. The mechanism by which the reduction takes place starts with the rise in pH as the water passes through the limestone bed. Lead is least soluble at a pH of approximately 10.0. In this test the pH was raised to approximately 9.5 by contact with the limestone. As the lead comes out of solution it is adsorbed onto the surface of the limestone.

3. The **3/8" limestone** was the least successful media tested for reducing the lead concentration. As with the Activated Alumina the lead in the effluent increased over the duration of the test starting from approximately 230 ppb to over 400 ppb. While the pH did increase through the 3/8" limestone bed the total surface area available for adsorption of lead within the 3/8" bed is significantly reduced over that of the fine grained limestone. The fine grained limestone has a surface area of approximately 2,000 times that of the 3/8" limestone in a given volume. Given these test results 3/8" limestone is not recommended for this site.

Sample	Liters processed	Bed Volumes	Lead (ppb)	Flow Rate (ml/min)	EBCT (min)	EBCT Test Average
Wurtsboro Mine Site						
Treatability Study Results						
Untreated pH = 5.77			536.4			
Activated Alumina						
3 Liters	3	38.6	9.18	36.72	6.32	
8 Liters	8	102.8	40.24	38.63	6.01	
13 Liters	13	167.1	43.78	36.92	6.29	
21 Liters	21	269.9	48.68	37.50	6.19	
39 Liters	39	501.3	94.58	37.85	6.13	6.19
Effluent pH = 9.74						
Limestone #16 / #60						
3 Liters	3	54.4	0.45	29.84	7.12	
8 Liters	8	145.0	0.39	23.39	9.08	
11 Liters	11	199.3	1.58	16.76	12.67	
17 Liters	17	308.1	0.45	17.79	11.94	
35 Liters	35	634.3	0.48	17.52	12.12	10.59
Effluent pH = 9.53						
3/8" Limestone						
Untreated - Total Lead			517.2			
Untreated Dissolved Lead			475.4			
3 Liters	3	6.7	231.9	84.99	10.08	
8 Liters	8	17.8	254.7	100.00	8.57	
21 Liters	21	46.8	355.7	104.17	8.22	
39 Liters	39	86.9	383.5	116.28	7.37	
70 Liters	70	155.9	410.4	90.91	9.42	8.73
70 Liters - Dissolved Lead	70	155.9	304.3	90.91	9.42	
Effluent pH = 9.74						
Minimum Detection Limit			0.12			

Recommendations

Given the test results it is clear that the media of choice is the fine grained limestone for the reduction of lead in the water. We have plotted the media on a grain size curve and have determine the hydraulic conductivity to be on the order of 1,000 – 3,000 gpd/ft^2. We believe it is possible to design and install an infiltration trench that allows the mine effluent to flow by gravity through the media and reduce lead to discharge levels. We would be happy to discuss this further if you so desire.

Attachments

1. *Reducing Lead and Selenium from Drinking Water Using Limestone-Based Material;* A Thesis present to the Department of Chemistry, Western Kentucky University; Sindhu Tumati, May 2012.

2. Laboratory results from Alpha Analytical dated December 31, 2015

3. Laboratory results from Alpha Analytical dated January 11, 2016

We trust this report is fully responsive to your request. If you have any questions regarding this matter please contact the writer.

Very truly yours
Ground/Water Treatment & Technology, LLC

Robert Kunzel
President and CEO

ATTACHMENT 1

REDUCING LEAD AND SELENIUM FROM DRINKING WATER USING
LIMESTONE-BASED MATERIAL

A Thesis
Presented to
The Faculty of the Department of Chemistry
Western Kentucky University
Bowling Green, Kentucky

In Partial Fulfillment
Of the Requirements for the Degree
Master of Science

By
Sindhu Tumati

May 2012

REDUCING LEAD AND SELENIUM FROM DRINKING WATER USING LIMESTONE-BASED MATERIAL

Date Recommended 5 - 3 - 2012

Cathleen J. Webb

Dr. Cathleen Webb, Director of Thesis

Dr. Bangbo Yan

Dr. Rui Zhang

Kinchel C. Doerner 13 -June - 2012

Dean, Graduate Studies and Research   Date

I dedicate this thesis to my parents, Prabhakar Reddy and Veerabhadra who are a great inspiration to me. I also dedicate this work to my behalf Sandeep Cheekati, who helped me greatly in everything to complete my Masters.

ACKNOWLEDGMENTS

I would first like to thank my research advisor, Dr. Cathleen Webb. Without her support this research would not have been possible. I am thankful to her for the guidance, knowledge and patience in assisting me throughout my research. Her understanding, personal guidance and support during some tough times at Western Kentucky University have really encouraged me to strive for the best in my life. As the Head of the department, she gave me continuous support throughout the Masters program.

I would also like to thank Ms. Pauline Norris for teaching me ICP for my work at the Advanced Materials Institute. I would like to thank Dr. John Andersland for his support in using the SEM. I would also like to thank Dr. Yan for his guidance in using XRD. I would also like to thank Dr. Rui Zhang for helping in my thesis defense.

I wish to thank the Department of Chemistry and the faculty and staff for helping me maximize my potential and knowledge in various fields. I am extremely thankful to the Advanced Material Institute at WKU for all the support and knowledge. I would also like to thank my friends at WKU for making my stay a wonderful one and also for supporting me during challenging times. I wish all the best to them.

Finally, I dedicate my thesis to my parents for their continuous support throughout my life.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF ABBREVIATIONS AND SYMBOLS

MCLG   Maximum Contaminant Level Goal

ppb   Parts Per Billion

ppm   Parts Per Million

ICP   Inductively Coupled Plasma

SEM   Scanning Electron Microscopy

XRD   X-Ray Diffraction

REDUCING LEAD AND SELENIUM FROM DRINKING WATER USING

LIMESTONE-BASED MATERIAL

Sindhu Tumati   May 2012   56 pages

Directed by: Dr. Cathleen Webb, Dr. Bangbo Yann, Dr. Rui Zhang

Department of Chemistry   Western Kentucky University

Contamination of drinking water with metals is a major problem facing many areas of United States and the World. There is a need for an inexpensive remediation technology for the removal of metals in drinking water that can be applied to small rural water systems. This research will focus on the development of a process for removal of select metals from drinking water by limestone-based material. Metals in drinking water considered for this research include lead and selenium. Limestone-based material has demonstrated the potential to reduce select metals (lead, cadmium and arsenic) in drinking water, with the additional benefit of low-cost disposal of a stable waste product in ordinary landfills.

Earlier research by the principal investigators using limestone-based material for drinking water treatment has clearly shown that this material can achieve metals removal of greater than 90 percent. This project will investigate techniques to improve removal efficiency of limestone-based material through adsorption and precipitation. This research will assist in the development of a granular adsorbent product that will remove metals and that can be manufactured and sold for use at the drinking water source, at point-of use, or at point-of entry.

Limestone was coated with Iron and its removal efficiency is compared with the uncoated limestone. Uncoated limestone was effective in removing lead completely from drinking water and iron-coated limestone was effective in removing selenium completely from drinking water. Effect of pH on removal of metals was also studied. Limestone is readily available and its use for metals removal is relatively inexpensive. The technology can be applied to small, rural water systems. Benefits of this research will include a low-cost treatment technology for source reduction that will reduce select metals to below drinking water standards.

I. INTRODUCTION

"Numerous metals have received attention as both environmental contaminants and potential toxicological hazards. For example, heavy metals like arsenic, cadmium, and lead are extensively distributed in the environment" (Chappell, et al, **1997**). Human activities have altered the natural distribution of these metals in the environment, which leads to the elevated concentration levels of these metals in drinking water. "The occurrence of arsenic, cadmium and lead and other metals in drinking water is an important pathway of potential exposure for citizens of the United States and many other nations in the world" (Barry Ryan, et al, **2000**). This research will focus on the removal of lead and selenium from drinking water.

As trace elements, some heavy metals (copper, selenium, zinc) are essential to the human body to maintain a healthy metabolism. However, at high concentrations, they lead to poisoning (Lobinski, et al, **1997**). To a small extent they can enter the human body through drinking water, food and air. In high concentrations, they tend to bioaccumulate in the body. Bioaccumulation means an increase in the concentration of a chemical in a biological organism over time, compared to the concentration of the chemical in the environment. Heavy metals can enter into water supplies by industrial and consumer wastes.

For this reason the US-EPA (United States Environmental Protection Agency) has established standard values for the contaminants allowed in drinking water as shown in table 1. Below this level, these contaminants are not considered harmful.

1

Table 1. Drinking water standards for some common heavy metals

Contaminant	MCLG (mg/L)
Arsenic	0.010
Lead	0.015
Selenium	0.05
Cadmium	0.005
Zinc	5
Copper	1.3

A. Lead:

"Lead is the commonest of the heavy elements and accounts for 13 mg/kg of the earth's crust. Lead is used in industries the production of lead acid batteries, solders, alloys, cable sheathing, pigments, rust inhibitors, ammunition, glazes, plastic stabilizers and many more" (*Lead—environmental aspects*. Geneva, World Health Organization, 1989 (Environmental Health Criteria, No. 85)). Lead is present in smaller quantities in tap water as a result of its dissolution from natural sources like household plumbing systems in which the pipes, solders and fittings contains lead. PVC pipes also contain some lead compounds that can be leached from them and result in higher concentrations of lead in drinking-water.

The amount of lead leached and dissolved in water from the plumbing system depends on different factors, including the presence of chloride and dissolved oxygen, pH, temperature, water softness, and standing time of the water. Acidic water is most suitable for lead as a solvent (Schock, et al, **1989**). The concentrations of lead in drinking water can be reduced by adding lime and also by adjusting the pH from <7 to 8-9 (Sherlock, et al, **1984**).

Prepared food in containers also contains small but a significant amount of lead. Lead content is increased when the water used for cooking or the cooking utensils contain lead, or the food, especially if acidic, has been stored in lead-soldered cans. The intake of lead from lead-soldered cans has been reduced as the use of lead-free solders became more widespread in the food processing industry (Galal-Gorchev, et al, **1991**).

A.1. Effect in humans:

Lead is a cumulative general poison. Infants, children up to 6 years of age, the fetus, and pregnant women are the most susceptible to lead and will have adverse health effects. It will mainly affect the central nervous system. Signs of acute intoxication include dullness, restlessness, irritability, headaches, muscle tremor, abdominal cramps, kidney damage, hallucinations, and loss of memory. "Encephalopathy occurs at blood levels of 100–120 µg/dl lead in adults and 80 – 100 µg/dl lead in children. After 1–2 years of exposure, muscle weakness, gastrointestinal symptoms, lower scores on psychometric tests, disturbances in mood, and symptoms of peripheral neuropathy were observed in occupationally exposed populations at blood lead levels of 40–60 µg/dl" (Campbell, et al, **1977**).

The pE-pH diagram represents the Nernst equation as a function of pH. The pE-pH diagram for lead is shown in figure 1 (Brookins, et al, **1988**). The upper dotted line represents the oxidation boundary of water and the bottom dotted line represents the reducing boundary of water. Drinking water has a pH range from 6-8. In this range, lead mainly exists as Pb[2+] ion. When the pH is increased to 7-9, lead forms lead hydroxide (PbOH[+]) and eventually precipitates as $Pb(OH)_2$.

Pb (JNC-TDB/GWB)
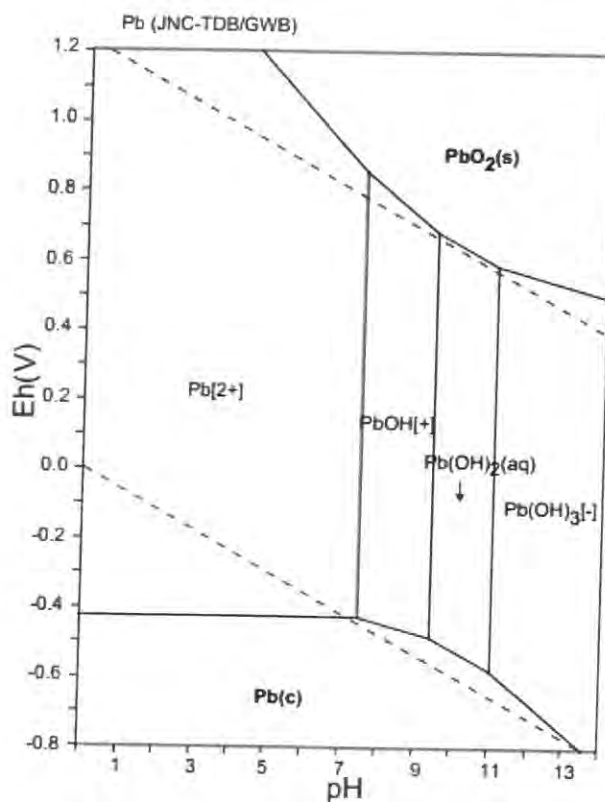
Figure 1. pE-pH diagram for lead (Brookins, et al, **1988**)

B. Selenium:

"Selenium is present in the earth's crust, sometimes in association with sulfur-containing minerals. The levels of selenium in groundwater and surface water ranges from 0.06 to about 400 µg/litre" (Smith, et al, **1937**). Selenium (Se) is recognized as an

4

essential micronutrient in most species, including humans. It is an important integral component of glutathione peroxidase (GSH-Px) and also has a function in detoxification of peroxides. Selenium also exhibits some inhibitory effect on carcinogenesis. Different health problems can arise with excess levels of selenium and as well as with a deficiency of selenium with a narrow margin between its essential and toxic actions.

"Foodstuffs constitute the main source of selenium for the general population. Daily dietary intake of selenium varies according to geographical area, food supplies, and the dietary habits. Recommended daily intakes have been set at 1.7 µg/kg of body weight in infants and 0.9 µg/kg of body weight in adults" (National Research Council. *Recommended dietary allowances*, 10th ed. Washington, DC, National Academy Press, 1989). Most of the drinking-water contains much less than 10 µg/litre, except in certain seleniferous areas. Total Selenium levels in the environment range from 0.1-400 µg/L in natural waters, to 0.06-1.8 ng/g in soils and a few nanograms per cubic meter in the atmosphere. The accumulation of total selenium depends on the environmental factors, and it is affected by pH (Selenium Concentrations in Natural and Environmental Waters, *Chem. Rev.* **1997,** 97, 1979-2003).

B.1. Effects in humans:

In humans, few reports of signs of selenium deficiency are available. It can be a factor in endemic cardiomyopathia (Keshan disease) and also possibly in joint and muscle disease (*Selenium.* Geneva, World Health Organization, 1987 (Environmental Health Criteria, No.58)). Acute oral doses of selenium compounds cause symptoms like nausea, diarrhoea, abdominal pain, chills, tremor, numbness in limbs, irregular menstrual bleeding, and marked hair loss (Sioris, et al, **1980**).

5

The pE-pH diagram for selenium is shown in figure 2 (Brookins, et al, **1988**). It shows the Nernst equation as a function of pH. From this diagram we can determine that, in typical drinking water, selenium will exist as a mixture of anions, $HSeO_3[-]$ and/or $SeO_4[2-]$. When the pH is increased, it will still exist as an anion ($SeO_4[2-]$) or possibly as $SeO_3[2-]$. These anions can chemisorb to the iron on the surface of the limestone through oxygen; hence it should be more easily removed with the iron-coated limestone than uncoated limestone.
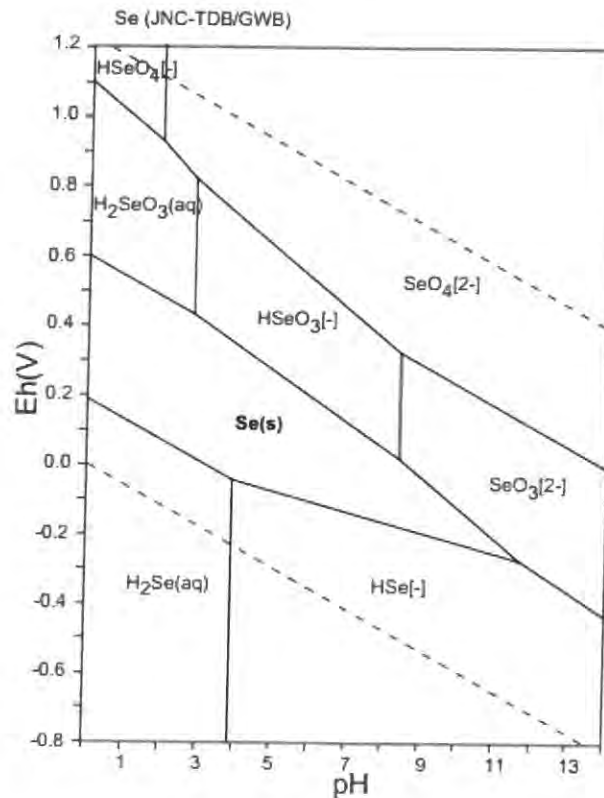
Figure 2. pE-pH diagram for selenium (Brookins, et al, **1988**)

A variety of treatment processes have been examined for heavy metal removal. Major technologies include coagulation/precipitation (McNeill, et al, **1997**), membrane separation (Waypa, et al, **1997**), ion exchange, and adsorption (Dambies, et al, **2004**),

reverse osmosis. Most of these techniques are expensive and have their own disadvantages. Among all these techniques adsorption is cost-effective and advantageous. Effectiveness of adsorption-based methods depends primarily on the adsorbent (granular media) used.

Different granular media can be used for removal of heavy metals from drinking water. Methods include granular activated alumina and granular activated carbon (Pattanayak, et al, **2000**) and limestone. These materials are less effective than iron oxides for arsenic adsorption. Limestone is used to remove many heavy metals like arsenic, selenium and lead (Mercedesdiaz-Somano, et al, **2004**). Several iron-based granular materials have been developed for the removal of heavy metals. They include iron oxide coated sand, sulfur-modified iron and granular ferric hydroxide (Zhimang, et al, **2005**).

Heavy metal adsorption or precipitation onto plain limestone is generally considered to be minimal, so it is not typically applied for water treatment. Literature has already shown that the adsorption on to the limestone can be increased significantly by treatment with various iron compounds (Reed, et al, **2000**). It is likely that some iron compounds produced by the treatment increase the surface area of the limestone, resulting in the enhanced removal.

Limestone properties:

Limestone has a very heterogeneous surface and has good buffering capacity. Uncoated limestone is shown in figure 3. Pretreatment of the sample pH is not necessary with the use of limestone. There is no need to recycle the limestone as disposal is also

easy. Limestone can be disposed in cement and is not leachable. Consequently, we propose to reduce heavy metals in drinking water using limestone as the base material.

Figure 3. Uncoated Limestone

The objective of this study is to develop and characterize a granular material that can be used for effective drinking water treatment. The heavy metals we are focusing on include lead and selenium. The base material used is limestone. We will also compare the effectiveness of iron-coated limestone and plain (uncoated) limestone to reduce heavy metals in drinking water. Here, the iron acts to increase the surface area of the limestone and, thereby, increases the capacity to remove metals. After treatment with limestone, water samples were analyzed with Inductively Coupled Plasma (ICP). This material was characterized by scanning electron microscope (SEM) and X-ray diffraction (XRD) technique. The ability of the media to remove heavy metals was evaluated through both batch and kinetic studies. The effect of pH on the removal was also studied in batch experiments.

II. EXPERIMENTAL SECTION

A. Chemicals and Materials:

Deionized water, obtained from a Nano Pure system was used throughout this work. Commercially available limestone (#16/60 limestone, Pete Lien and Sons, LaPorte, CO) was used on-site. Earlier studies were conducted with limestone that was ground and sieved. The commercially available limestone, commonly used in chicken feed, was found to be quite efficient.

A.1. Preparation of Iron-coated limestone:

A sample of 100g of limestone was placed into a round bottom flask. A 100mL solution of 0.1M iron (III) chloride ($FeCl_3$) was then placed in the flask. The flask was placed on a shaker. After 24 hours, a few drops of concentrated sodium hydroxide is added to the solution to help precipitate iron. The granules were then rinsed with deionized water and air dried. Iron-coated limestone after drying is shown in figure 4.

Figure 4. Iron-coated Limestone

A.2. Preparation of solutions:

Standard solutions (1000 ppm) of lead and selenium were purchased from Inorganic Ventures. All other solutions were prepared from these standards.

Preparation of 10ppm lead solution:

An aliquot of 1mL of the standard lead solution was place in a 100mL volumetric flask and the volume is made up to 100mL with deionized water.

Preparation of 50ppb lead solution:

An aliquot of 5mL of the 10ppm lead solution is taken into a 1000mL volumetric flask and the volume is made up to 1000mL with deionized water.

Preparation of 100ppb lead solution:

An aliquot of 10mL of the 10ppm lead solution is taken into a 1000mL volumetric flask and the volume is made up to 1000mL with deionized water.

Preparation of 200ppb lead solution:

An aliquot of 20mL of the 10ppm lead solution is taken into a 1000mL volumetric flask and the volume is made up to 1000mL with deionized water.

Preparation of 10ppm selenium solution:

An aliquot of 1mL of the standard selenium solution is taken into a 100mL volumetric flask and the volume is made up to 100mL with deionized water.

Preparation of 50ppb selenium solution:

An aliquot of 5mL of the 10ppm selenium solution is taken into a 1000mL volumetric flask and the volume is made up to 1000mL with deionized water.

<u>Preparation of 100ppb selenium solution:</u>

An aliquot of 10mL of the 10ppm selenium solution is taken into a 1000mL volumetric flask and the volume is made up to 1000mL with deionized water.

<u>Preparation of 200ppb selenium solution:</u>

An aliquot of 20mL of the 10ppm selenium solution is taken into a 1000mL volumetric flask and the volume is made up to 1000mL with deionized water.

<u>B. Apparatus:</u>

A Burrell Wrist action shaker (model 75) was used to stir the solutions. The shaker is shown in figure 5. The shaker was kept on a speed of 1 cycle/sec and the solutions were stirred along with the limestone. Whatman cellulose nitrate membrane filters (0.45micron) were used to filter the solutions using a micro filtration syringe apparatus. A Fisher Scientific AB 15 pH meter was used to measure the pH of all the solutions. The pH meter is shown in figure 6. All the glassware is made of Pyrex glass.
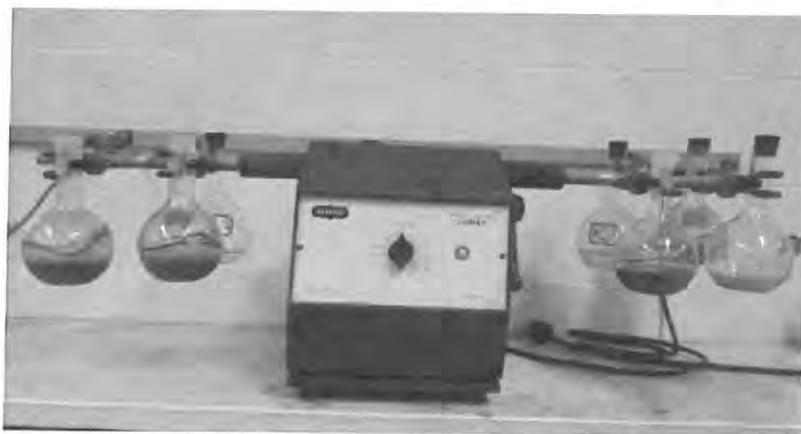
Figure 5. Burrell Wrist Action Shaker

All the samples were analyzed using Inductively Coupled Plasma (ICP). Microscopic studies of the limestone are done using Scanning Electron Microscopy and X-ray Diffraction. The ICP instrument is located at the Advanced Material Institute (AMI) lab, which is located in the Center for Research and Development, Western Kentucky University.

Figure 6. Fisher scientific AB 15 pH meter

C. Methods:

C.1. Kinetics:

Kinetic tests are conducted using a fixed amount of limestone (5g) and standard solution volumes (100mL) using different time intervals (30min, 1hr, 1.5hrs, 2hrs, 4hrs, 10hrs and 24hrs). Different concentrations of standard solutions (50, 100 and 200ppb) are used for this experiment. A volume of 100mL of each standard solution is placed in the round bottomed flask and kept in contact with coated and uncoated limestone (5g) for the different intervals of time.

C.2. Batch tests:

Batch tests are conducted using different amounts of limestone (5, 10, 20, 50 and 100g) with 100mL of the prepared standard solutions for both Lead and Selenium. The limestone and the solution are placed in the round bottomed flask and stirred for 5hours for Selenium and 30 minutes for Lead.

C.3. pH studies:

The effect of pH on the removal of the heavy metals is also studied by using solutions of different initial pH. Standard solutions of lead (100ppb) and selenium (100ppb) with varying initial pH (pH 5, 6, 7, 8 and 9) were placed and kept in contact with the limestone on the shaker. The initial pH of the standard solutions was adjusted with 1M sodium hydroxide. The final pH of the solutions after treatment with the limestone was also measured.

C.4. Sample collection and analysis:

After treatment with the limestone, the solutions are filtered using 0.45 micron Whatman cellulose nitrate membrane filters. All the samples are collected in glass bottles and analyzed using Inductively Coupled Plasma (ICP).

C.5. Calibration:

Samples are analyzed by ICP using the 1000ppm standard solutions of lead and selenium for the calibration of the instrument. Different concentrations of standard solutions (blank, 5, 10, 20, 30, 40 and 50ppb) are prepared with deionized water. The instrument is calibrated and the samples are loaded onto the auto sampler tray.

C.6. Sample introduction:

The nebulizer is used in ICP to inject the samples. This converts liquids into an aerosol, and the aerosol is swept into the plasma to create the ions. The plasma used in an ICP is made by using argon gas.

C.7. Auto session:

Flush time is kept for 80 seconds. The wavelength used for lead is 220.353nm and the wavelength for selenium is 196.026. To analyze the samples, first the water flow is started and the plasma is turned on. Auto session is turned on and calibration of the instrument is done first. As the instrument consists of an auto sampler, it will run automatically. It takes 3 minutes for each sample to run and analyze.

C.8. Microscopic studies

Microscopic studies of the limestone are done by using SEM (Scanning Electron Microscopy) and XRD (X-Ray Diffraction). Scanning electron microscopy uses beams of electrons and gives the information about the sample's surface topography and composition. SEM studies are done for the uncoated limestone, iron-coated limestone, uncoated limestone after the treatment with 100ppb lead solution and iron-coated limestone after the treatment with 100ppb selenium solution.

Scanning electron microscope consists of a sample holder, an electron column and an electron detector. Samples are held on cylindrical stubs using a carbon tape. An excess sample is removed by blowing compressed air on it. Stubs are placed in the sample

holder and analyzed using SEM. The sample holder contains four cylindrical mounts to hold the stubs.

X-Ray diffraction provides information about the crystal structure, chemical composition and physical properties of materials. The uncoated and the iron-coated limestone is ground, homogenized and analyzed to determine their composition using XRD. X-ray diffractometer consists of an X-ray tube, a sample holder and an X-ray detector.

III. RESULTS AND DISCUSSIONS

A. Effect of contact time:

The effect of contact time on the removal of lead and selenium was examined using a fixed amount of limestone (5g) and different time intervals. Different concentrations of standard solutions (50, 100 and 200ppb) were used to study the kinetics. Plain and iron-coated limestones are used for the comparison of their removal capacity.

The calibration curve for lead is shown in figure 7. The graph is plotted between concentration on X-axis and Intensity on Y-axis. Calibration is done with different concentrations (5, 10, 20, 30, 40 and 50) of lead standard solution. The R^2 value for the calibration curve is 0.999.

Figure 7. Calibration curve for Lead

The effect of time on removal of lead was studied through kinetic studies. The results for the kinetics experiment for 50ppb lead solution with 5 grams uncoated limestone is shown in table 2 and figure 8.

Table 2. Kinetics experiment with 50ppb lead solution using 5g uncoated limestone

Time (in minutes)	Concentration (in ppb) ($\pm$0.05)
2	ND
5	3.23
10	ND
15	4.55
20	4.37
25	2.36
30	4.87
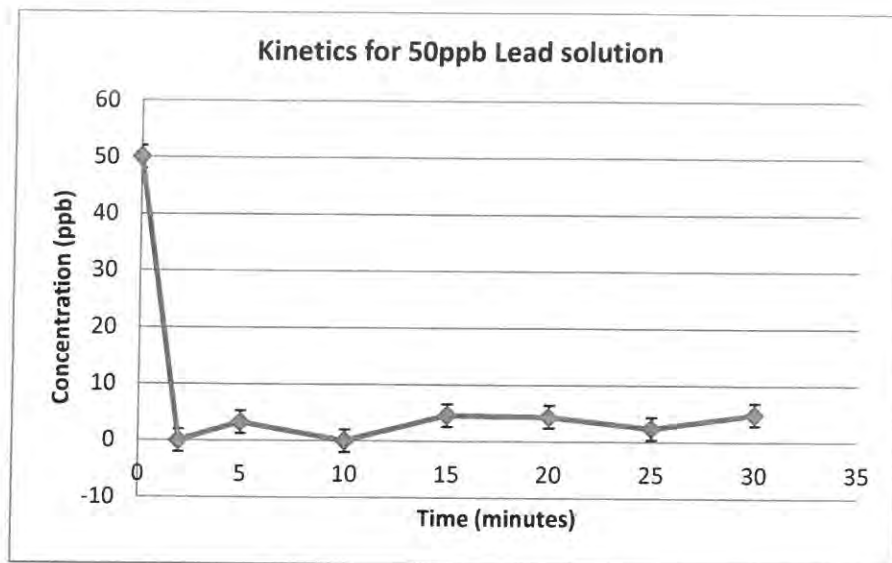
Figure 8. Kinetics experiment with 50ppb lead solution using 5g uncoated limestone

Lead has been reduced to below the drinking water standard of 15ppb within 3 minutes from an initial concentration of 50ppb, when treated with 5 grams of uncoated limestone.

The results for the kinetics experiment for 100ppb lead solution with 5 grams uncoated limestone is shown in table 3 and figure 9.

Table 3. Kinetics experiment with 100ppb lead solution using 5g uncoated limestone

Time (in minutes)	Concentration (in ppb) (± 0.05)
2	0.59
5	0.42
10	0.43
15	0.10
20	0.21
25	0.13
30	0.13
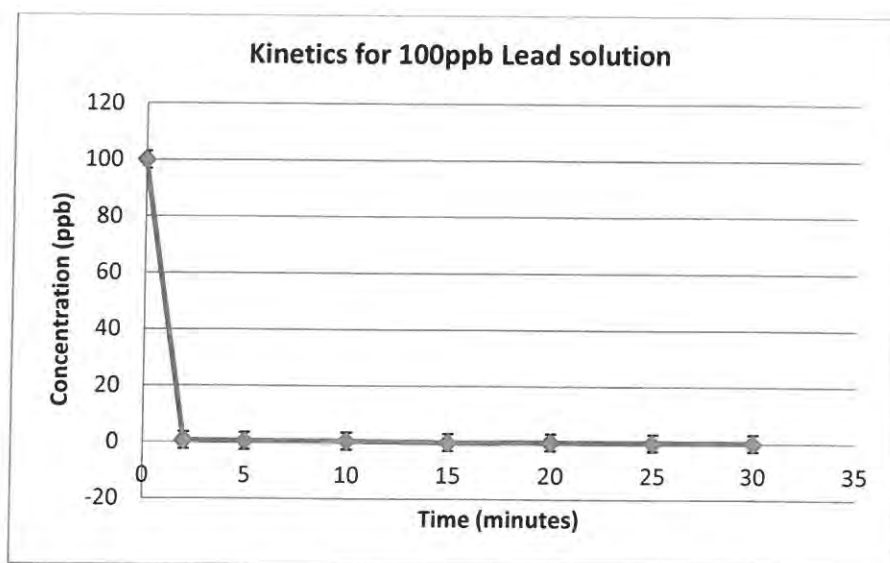
Figure 9. Kinetics experiment with 100ppb lead solution using 5g uncoated limestone

Lead has been reduced to below the drinking water standards within 30 minutes from an initial concentration of 100ppb, when treated with 5grams of uncoated limestone.

19

15 ppb = 3M
100 ppb = 30M

The results for the kinetics experiment for 200ppb lead solution with 5 grams uncoated limestone is shown in table 4 and figure 10.

Table 4. Kinetics experiment with 200ppb lead solution using 5g uncoated limestone

Time (in minutes)	Concentration (in ppb) (± 0.05)
2	0.25
5	0.49
10	0.32
15	0.18
20	0.01
25	0.59
30	0.09
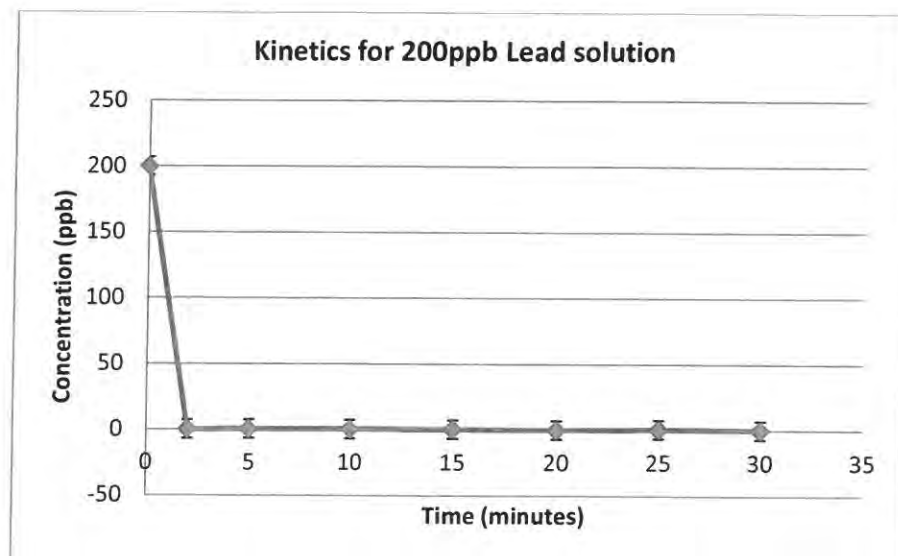
Figure 10. Kinetics experiment with 200ppb lead solution using 5g uncoated limestone

Lead has been reduced to below the drinking water standards within 10 minutes from an initial concentration of 200ppb, when treated with 5 grams of uncoated limestone.

200ppb = 100~

The results for the kinetics experiment for 50ppb lead solution with 5 grams Iron-coated limestone is shown in table 5 and figure 11.

Table 5. Kinetics experiment with 50ppb lead solution using 5g iron coated limestone

Time (in hours)	Concentration (in ppb) ($\pm$0.05)
0.5	3.74
1	1.62
1.5	0.21
2	3.15
4	4.46
10	ND
24	ND
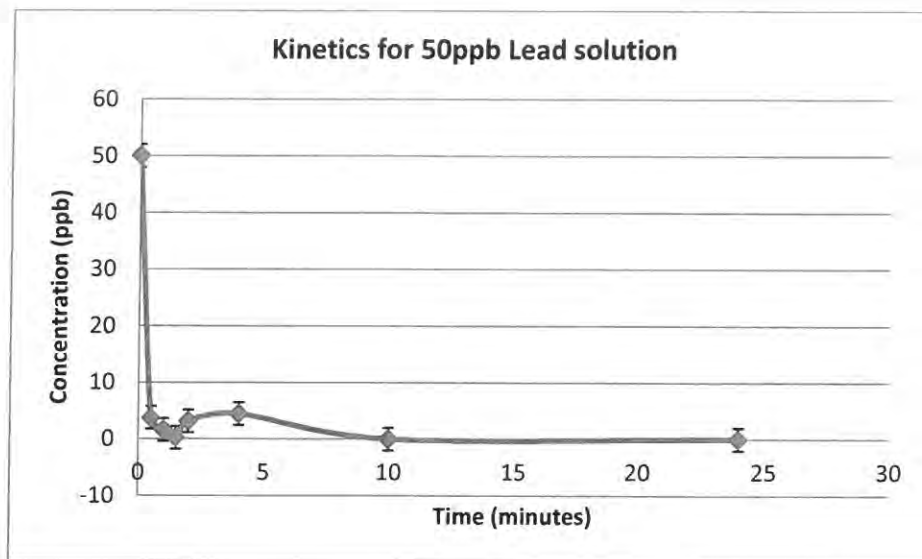
Figure 11. Kinetics experiment with 50ppb lead solution using 5g iron-coated limestone

Lead has been reduced to non-detect levels within 10 hours from an initial concentration of 50ppb, when treated with 5 grams of uncoated limestone. The uncoated limestone is more effective than the iron-coated limestone, because uncoated limestone removes lead completely within 30 minutes.

50ppb
= 10 hrs

The calibration curve for selenium is shown in figure 12. The R^2 value for the calibration of selenium is 0.999. The graph is plotted between concentration on X-axis and intensity on Y-axis. Calibration is done with different concentrations (10, 20, 30, 40 and 50) of selenium standard solution. The R^2 value for the calibration curve is 0.998.
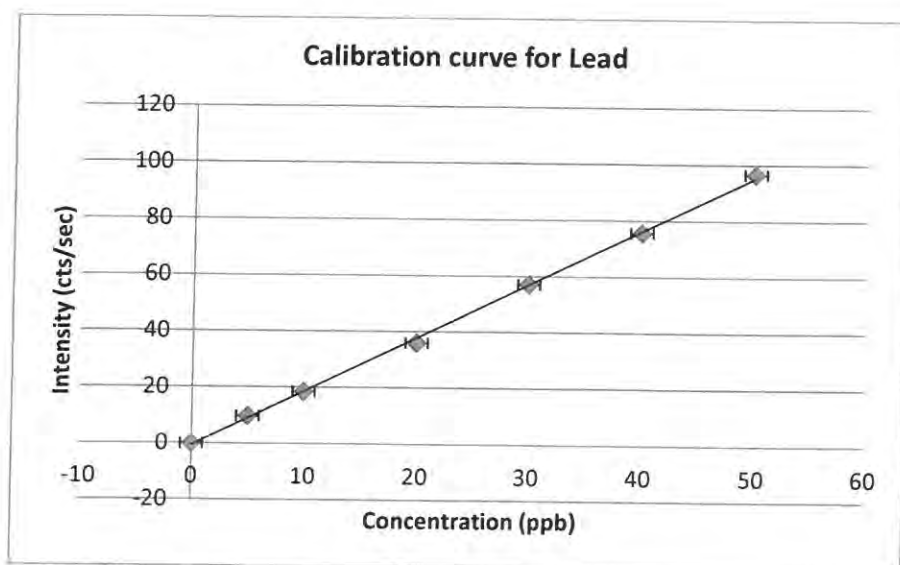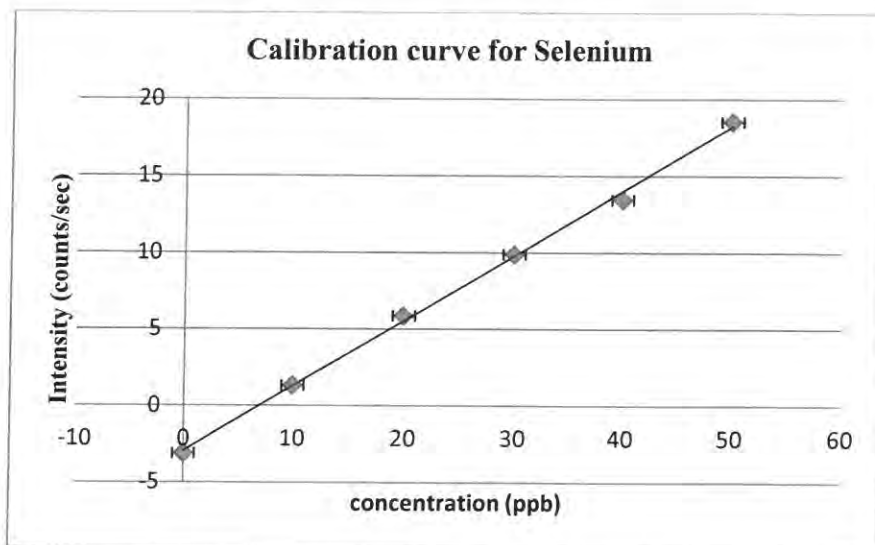

Figure 12. Calibration curve for selenium

The effect of time on removal of selenium was studied through kinetic studies. The results for the kinetics experiment for 50ppb selenium solution with 5 grams Iron-coated limestone is shown in table 6 and figure 13.

Table 6. Kinetics experiment with 50ppb selenium solution using 5g iron-coated limestone

Time (in hours)	Concentration (in ppb) (± 0.05)
0.5	0.30
1	1.06
1.5	0.76
2	1.03
4	0.80
10	0.12
24	2.57
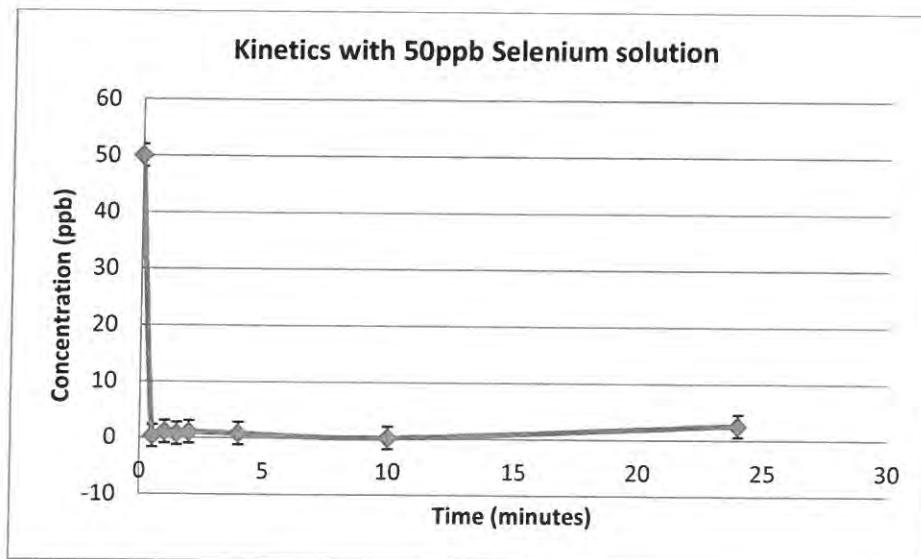
Figure 13. Kinetics experiment with 50ppb selenium solution using 5g iron-coated limestone

Selenium has been removed to less than the drinking water standard of 50ppb within 5 hours only. Removal of 99.4% of the selenium after treatment with 100mL solution of 50ppb concentration and 5 grams of iron-coated limestone occurred in 5 hours.

The results for the kinetics experiment for 100ppb selenium solution with 5 grams iron-coated limestone is shown in table 7 and figure 14.

Table 7. Kinetics experiment with 100ppb selenium solution using 5g iron-coated limestone

Time (in hours)	Concentration (in ppb) (±0.05)
0.5	9.63
1	10.0
1.5	9.56
2	10.5
3	8.96
4	7.48
5	12.0
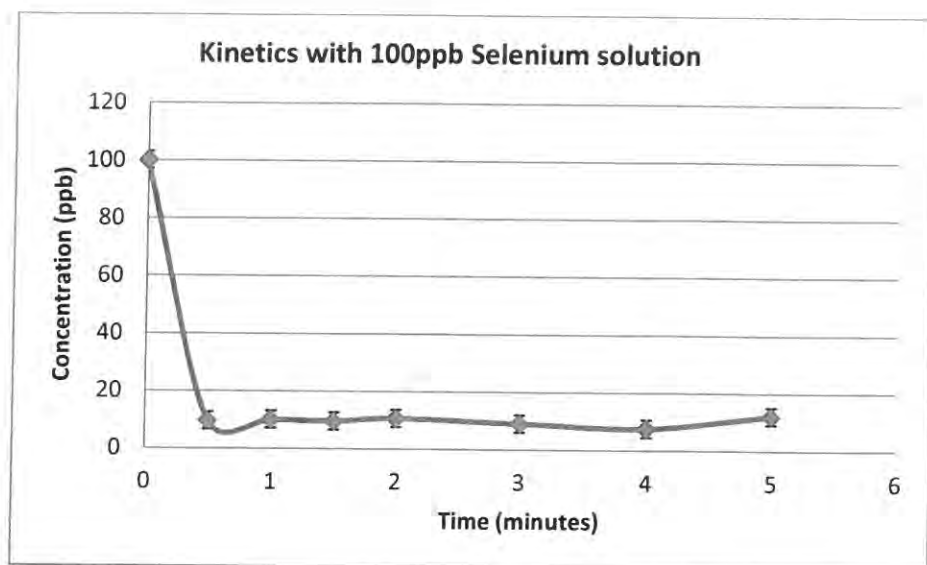
Figure 14. Kinetics experiment with 100ppb selenium solution using 5g iron-coated limestone

Selenium has been removed to less than its drinking water standard within 4 hours. It can be seen that 92.5% of the selenium was removed after treatment with 100mL solution of 100ppb concentration and 5grams of iron-coated solution.

The results for the kinetics experiment for 200ppb selenium solution with 5 grams iron-coated limestone is shown in table 8 and figure 15.

Table 8. Kinetics experiment with 200ppb selenium solution using 5g iron-coated
limestone

Time (in hours)	Concentration (in ppb) ($\pm$0.05)
0.5	19.99
1	16.71
1.5	14.86
2	18.15
3	10.86
4	14.97
5	23.35
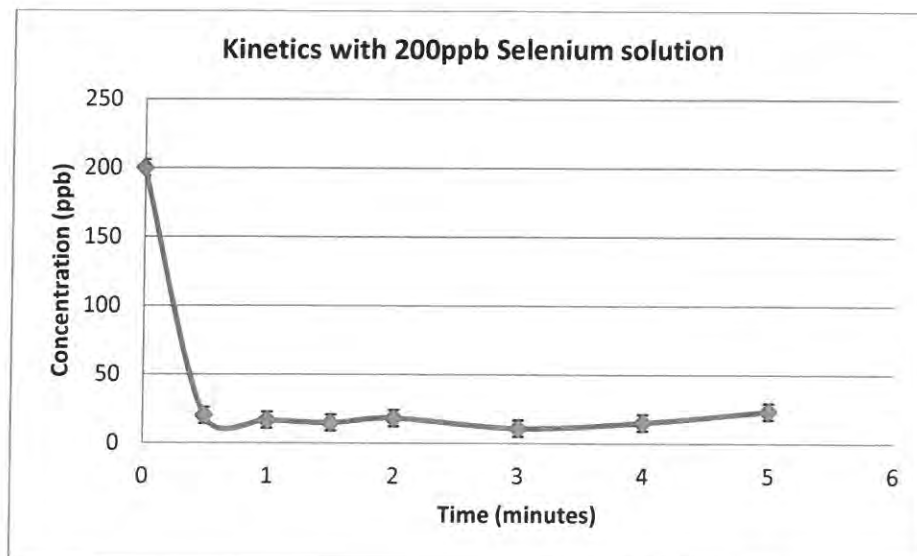
Figure 15. Kinetics experiment with 200ppb selenium solution using 5g iron-coated
limestone

Selenium has been removed to less than its drinking water standard within 3 hours with 94.6% of the selenium removed after treatment with 100mL solution of 200ppb concentration and 5grams of iron-coated solution.

The results for the kinetics experiment for 50ppb selenium solution with 5 grams uncoated limestone is shown in table 9 and figure 16.

Table 9. Kinetics experiment with 50ppb selenium solution using 5g uncoated limestone

Time (in hours)	Concentration (in ppb) ($\pm$0.05)
0.5	13.07
1	40.15
1.5	37.62
2	38.26
4	37.31
10	33.23
24	10.19
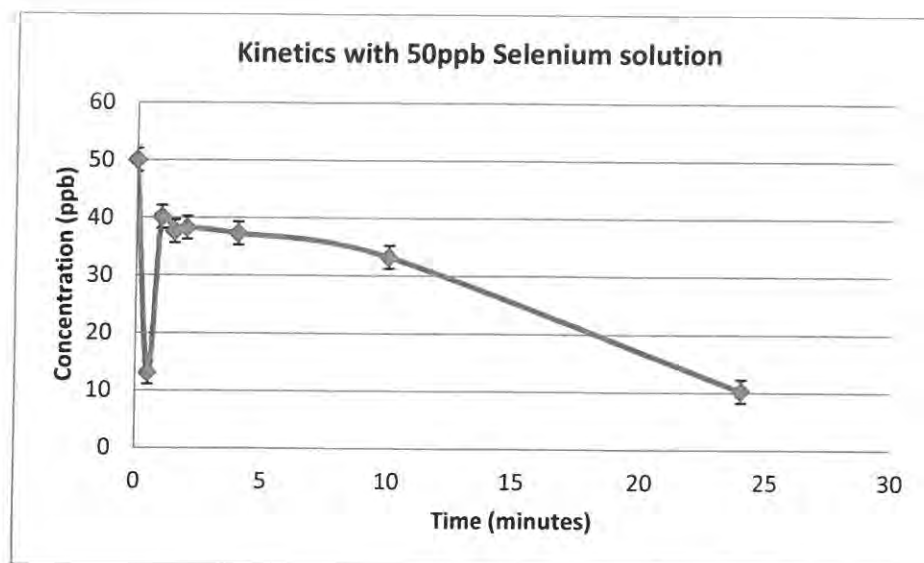
Figure 16. Kinetics experiment with 50ppb selenium solution using 5g uncoated limestone

In this experiment 5 grams of uncoated limestone removes 79.6% of the selenium from a 100mL solution of 50ppb concentration in 24 hours.

B. Effect of limestone amount:

The effect of limestone on the removal capacity was studied using different amounts of limestone (5, 10, 20, 50 and 100g) while the contact time is kept the same for all samples. The results are also compared with different concentrations (50, 100 and 200ppb) of lead and selenium standard solutions.

The effect of limestone on the removal of lead was studied using batch tests. The results for the batch test with 50ppb lead solution using uncoated limestone are shown in table 10 and figure 17.

Table 10. Batch test with 50ppb lead solution using uncoated limestone

Weight of limestone (grams)	Concentration (ppb) (± 0.05)
5	4.87
10	ND
20	2.58
50	0.36
100	0.72
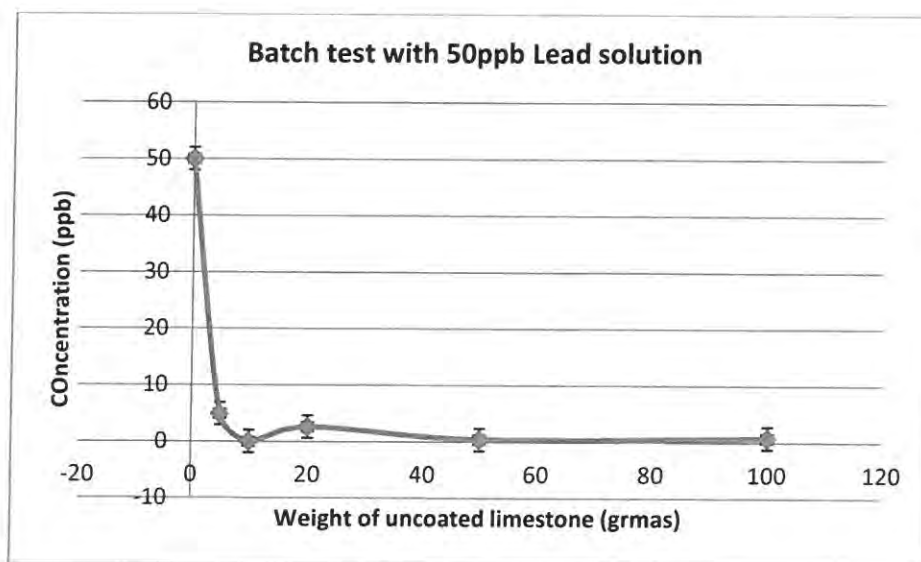
Figure 17. Batch test with 50ppb lead solution using uncoated limestone

Different amounts of plain limestone (5, 10, 20, 50 and 100g) are used to remove lead. A sample of 10grams of uncoated limestone efficiently removes lead completely from a solution of 50ppb concentration to non-detectable levels.

The results for the batch test with 100ppb lead solution using uncoated limestone is shown in table 11 and figure 18.

Table 11. Batch test with 100ppb lead solution using uncoated limestone

Weight of limestone (grams)	Concentration (ppb) ($\pm$0.05)
5	0.13
10	0.76
20	0.10
50	0.25
100	0.12
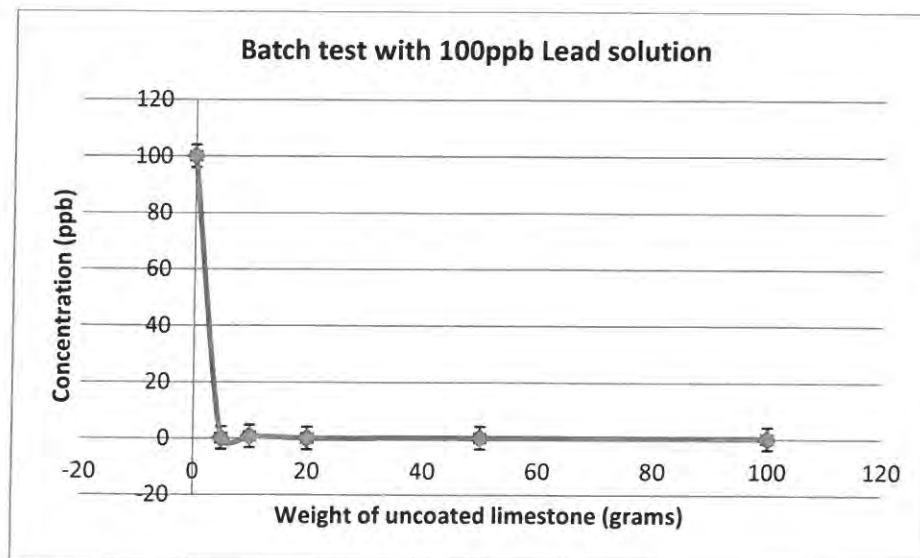
Figure 18. Batch test with 100ppb lead solution using uncoated limestone

Uncoated limestone efficiently removes lead from 100ppb solution. Twenty grams limestone is more efficient than the other amounts removing up to 99.8% of the lead from a 100mL solution of 100ppb concentration.

The results for the batch test with 200ppb lead solution using uncoated limestone is shown in table 12 and figure 19.

Table 12. Batch test with 200ppb lead solution using uncoated limestone

Weight of limestone (grams)	Concentration (ppb) ($\pm$0.05)
5	0.09
10	0.25
20	0.30
50	0.00
100	0.01
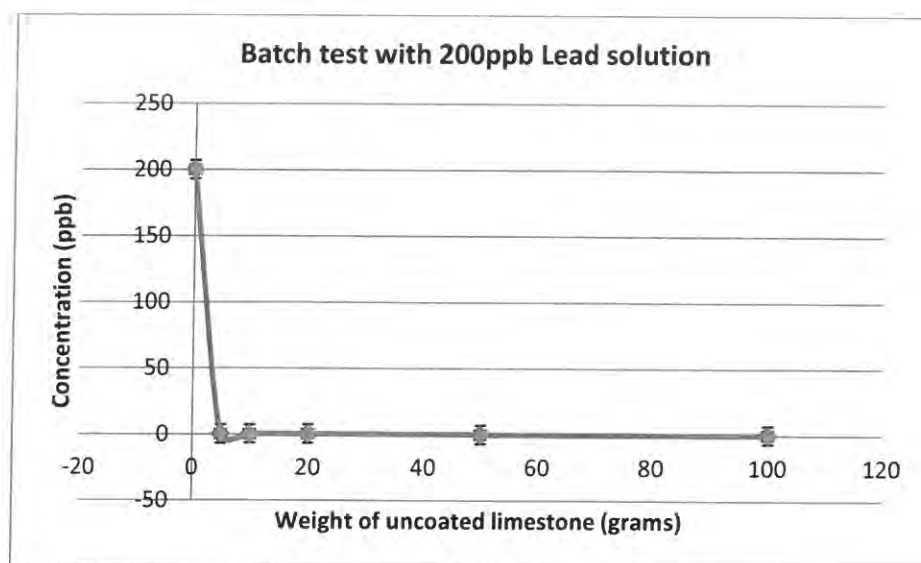
Figure 19. Batch test with 200ppb lead solution using uncoated limestone

In this experiment 50 grams of limestone removes 99.99% of lead efficiently from a 100mL solution of 200ppb concentration.

The effect of limestone on the removal of selenium was studied using batch tests. The results for the batch test with 100ppb selenium solution using uncoated limestone is shown in table 13 and figure 20.

Table 13. Batch test with 100ppb selenium solution using iron-coated limestone

Weight of limestone (grams)	Concentration (in ppb) (± 0.05)
5	12.02
10	6.02
20	4.07
50	4.86

33

100	5.12

Batch test with 100ppb Selenium solution
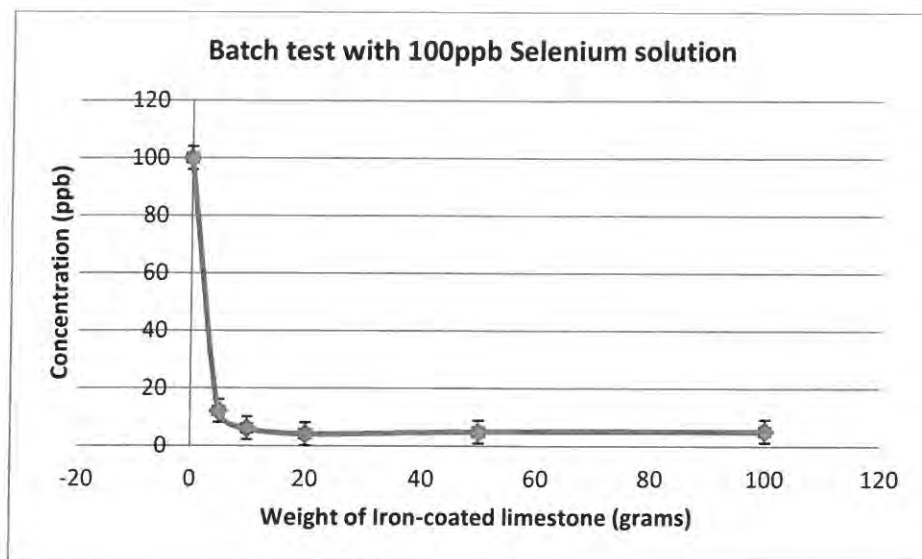
Figure 20. Batch test with 100ppb selenium solution using iron-coated limestone

Batch tests with limestone are very effective with all the amounts. It was found that 20 grams of iron-coated limestone is more effective for removal of selenium from a 100mL solution of 100ppb concentration, since this level removes 95.92% of the selenium from the solution.

The results for the batch test with 200ppb selenium solution using uncoated limestone is shown in table 14 and figure 21.

Table 14. Batch test with 200ppb selenium using Iron-coated limestone

Weight of limestone (grams)	Concentration (in ppb) (± 0.05)
5	23.3
10	10.5
20	5.99
50	5.01
100	4.20
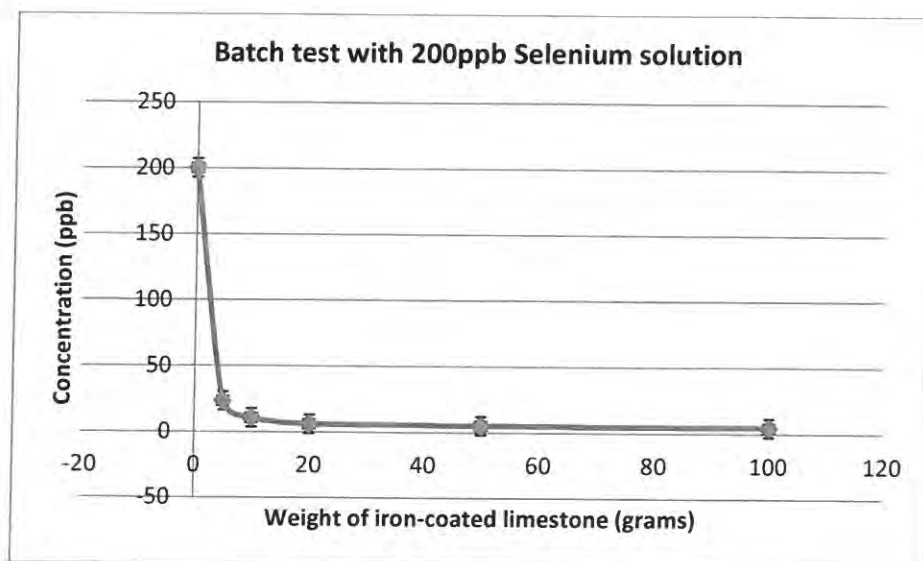
Figure 21. Batch test with 200ppb selenium solution using iron coated limestone

All the amounts of limestone are effective and removes selenium below its drinking water standard. A sample of 100 grams iron-coated limestone is most effective because it removes 97.9% of the selenium from the solution.

C. Effect of pH on Adsorption Capacity:

The effect of pH on the removal of metals is studied by adjusting the pH of the standard solutions. The pH was adjusted by using 1M sodium hydroxide (NaOH). The effect of pH on removal of lead is shown in table 15 and figure 18. The effect of pH on the removal of selenium is shown in table 16 and figure 19.

Table 15. Effect of pH using 100ppb lead solution with 5g uncoated limestone

Initial pH	% removal ($\pm$0.05)	Final pH
5	33.2	8.1
6	73.0	8.2
7	40.8	8.2
8	64.2	8.5
9	3.00	8.7
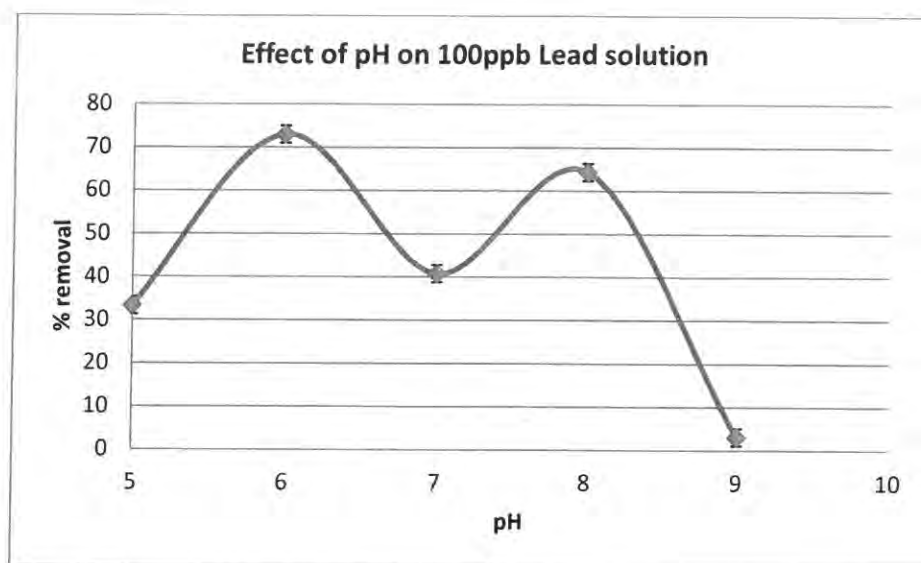
Figure 22. Effect of pH using 100ppb lead solution with 5g uncoated limestone

The pH 6 is most efficient at removing lead from a 100mL solution of 100ppb concentration with 73.0% of the lead removed when the pH was adjusted to 6. The initial pH of the 100ppb lead solution is 2.6. Final pH of the solutions after the treatment with uncoated limestone were measured and are shown in table 14. This buffering is expected as limestone is the base material.

Table 16. Effect of pH using 100ppb selenium solution with 5g iron-coated limestone

Initial pH	% removal ($\pm$0.05)	Final pH
5	85.5	8.3
6	94.6	8.3
7	90.1	8.4
8	90.4	8.6
9	89.4	9.0

37
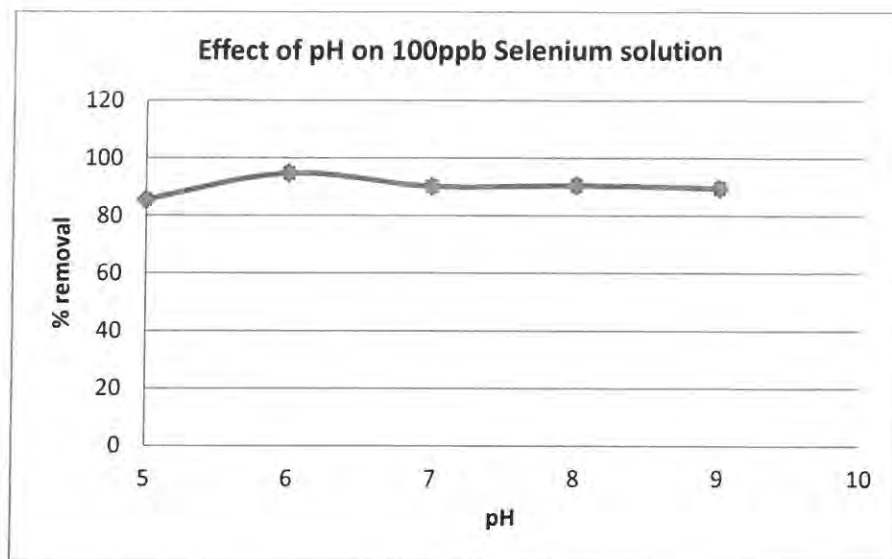
Figure 23. Effect of pH using 100ppb selenium solution with 5g iron-coated limestone

A pH 6 is most effective to remove selenium from a 100mL solution of 100ppb concentration. However, all tested pH conditions are also effective at removing selenium to below the drinking water standard. When the pH is adjusted to 6, selenium has a 94.7% removal.

D. Microscopic studies:

D.1. Scanning Electron Microscopy:

Scanning Electron Microscopic (SEM) images of the plain (uncoated) limestone, iron-coated limestone, plain limestone after treatment with 100ppb of lead solution and coated limestone after treatment with 100ppb of selenium solution are taken using Scanning Electron Microscopy. SEM images are shown in figures 24, 25, 26 and 27.
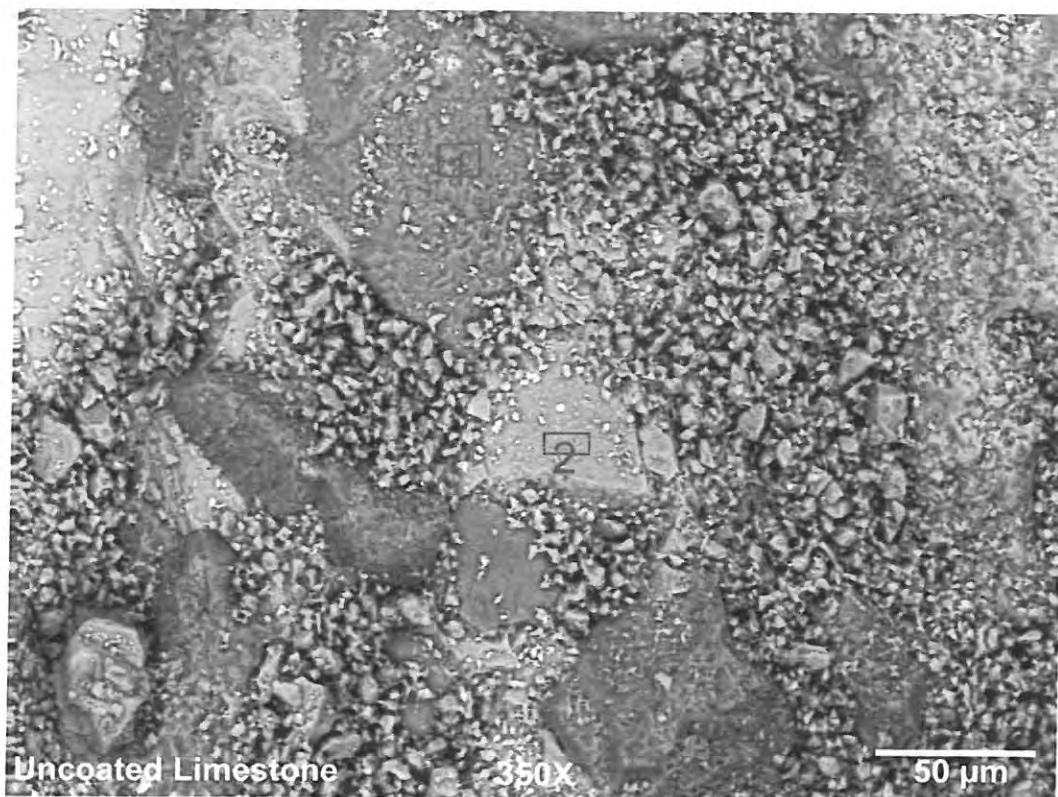
Figure 24. SEM image of uncoated limestone

Analysis Report:
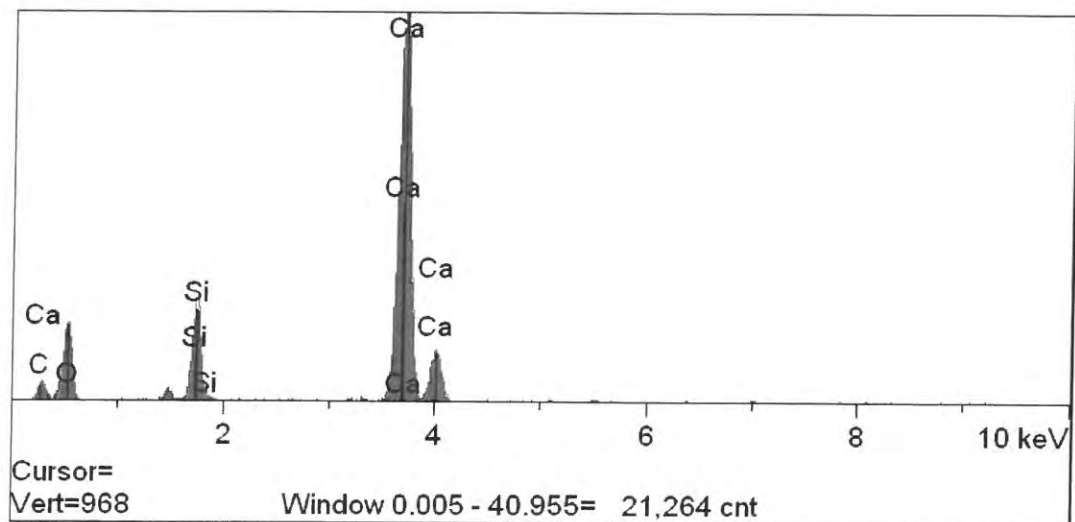
Table 17. SEM analysis of uncoated limestone

	Image-1		Image-2	
Elt.	Atomic %	Conc Wt%	Atomic %	Conc Wt%
C	5.07	3.03	9.88	5.71
O	66.35	52.80	66.43	51.13
Si	21.47	30.00	4.38	5.92
Ca	7.11	14.18	19.31	37.24
Total	100.00	100.00	100.00	100.00

The analysis of the SEM images of the uncoated limestone indicates that it is primarily $CaCO_3$ with small amounts of silicon (SiO_2).
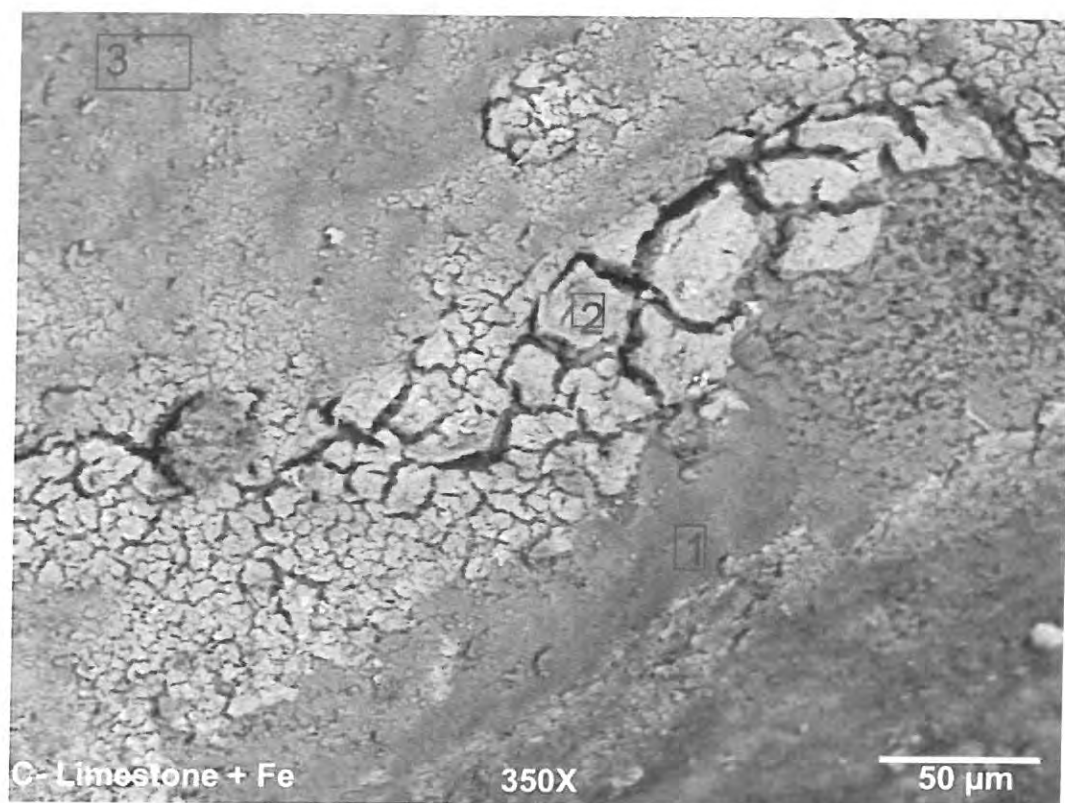
Figure 25. SEM image of iron-coated limestone

Analysis Report:
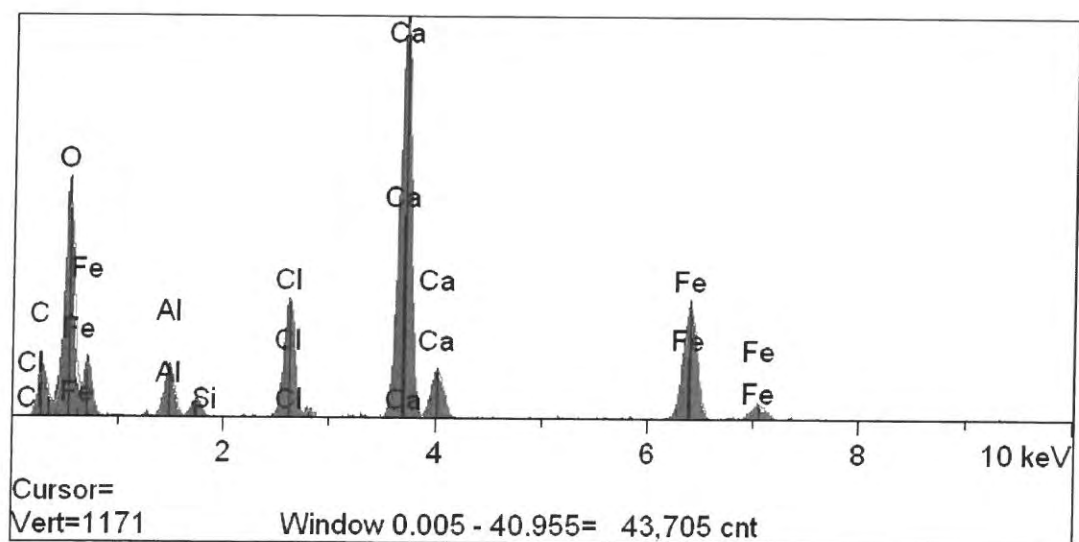
Table 18. SEM analysis of iron-coated limestone

Elt.	Image-1		Image-2		Image-3	
	Atomic %	Conc (wt.%)	Atomic %	Conc (wt.%)	Atomic %	Conc (wt.%)
C	20.01	13.01	18.17	10.89	16.34	10.23
O	65.46	56.67	63.38	50.58	67.36	56.17
Al	2.95	4.30	1.89	2.55	2.07	2.91
Si	0	0	0.55	0.77	0.29	0.42
Cl	0.47	0.91	2.41	4.27	0.72	1.33
Ca	9.90	21.46	8.80	17.59	11.61	24.24
Fe	1.21	3.65	4.80	13.37	1.61	4.70
Total	100.00	100.00	100.00	100.00	100.00	100.00

The analysis of SEM images of the iron-coated limestone indicates that iron is not distributed uniformly and is clearly on the surface of the limestone. The bands where iron is accumulated can be observed directly. The limestone particles also have some chloride probably due to the use of the iron chloride solution to prepare the iron-coated limestone.
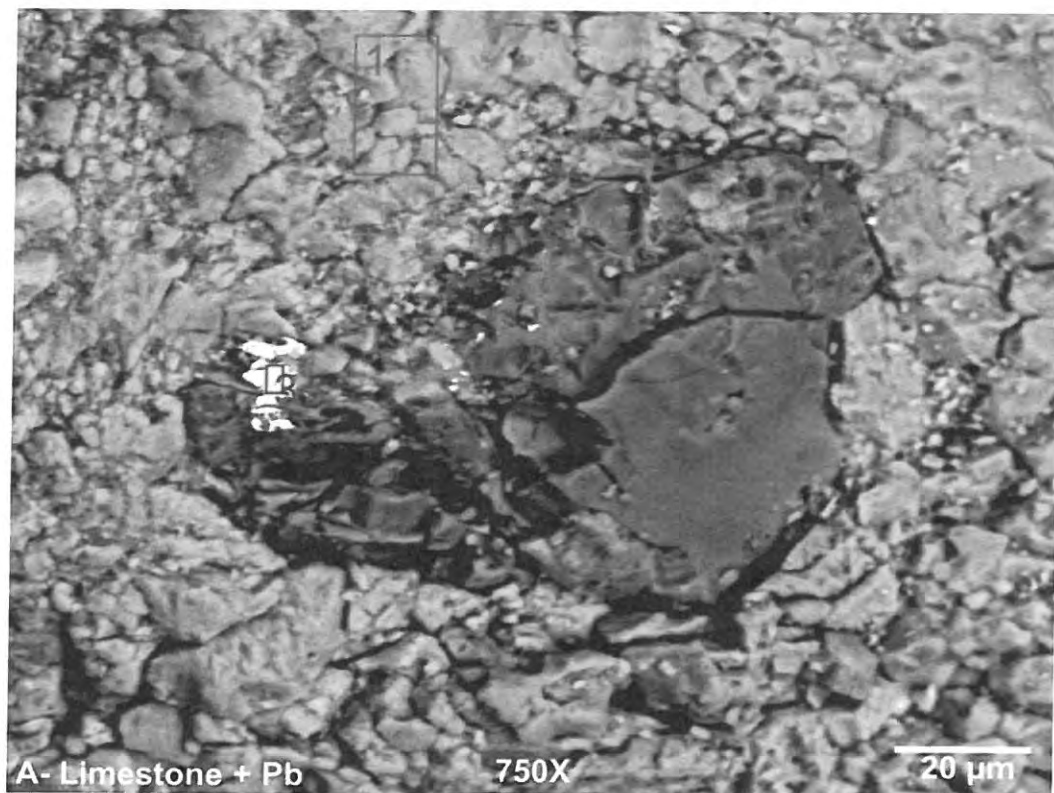
Figure 26. SEM image of the uncoated limestone after the treatment with 100ppb lead solution

Analysis Report:
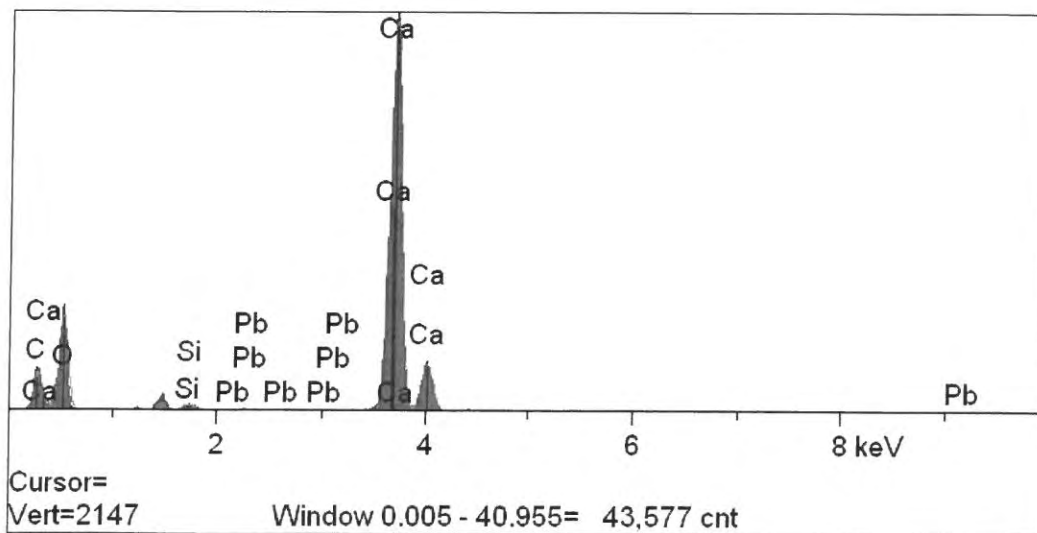
Table 19. SEM analysis of uncoated limestone after treatment with 100ppb lead solution

Elt.	Image-1		Image-2		Image-3	
	Atomic %	Conc Wt%	Atomic %	Conc Wt%	Atomic %	Conc Wt%
C	13.63	8.21	8.71	5.50	14.29	8.87
O	67.15	53.89	67.26	56.55	69.39	57.37
Si	1.25	1.77	20.08	29.64	0.38	0.56
Ca	17.97	36.13	3.95	8.32	15.91	32.95
Pb	0.00	0.00	0.00	0.00	0.02	0.26
Total	100.00	100.00	100.00	100.00	100.00	100.00

The analysis of the SEM image of the plain limestone after treatment with 100ppb lead solution indicates that the material does have small amounts of lead precipitated on the surface which actually can be seen as bright spots in image-3 in table 19 and figure 26.
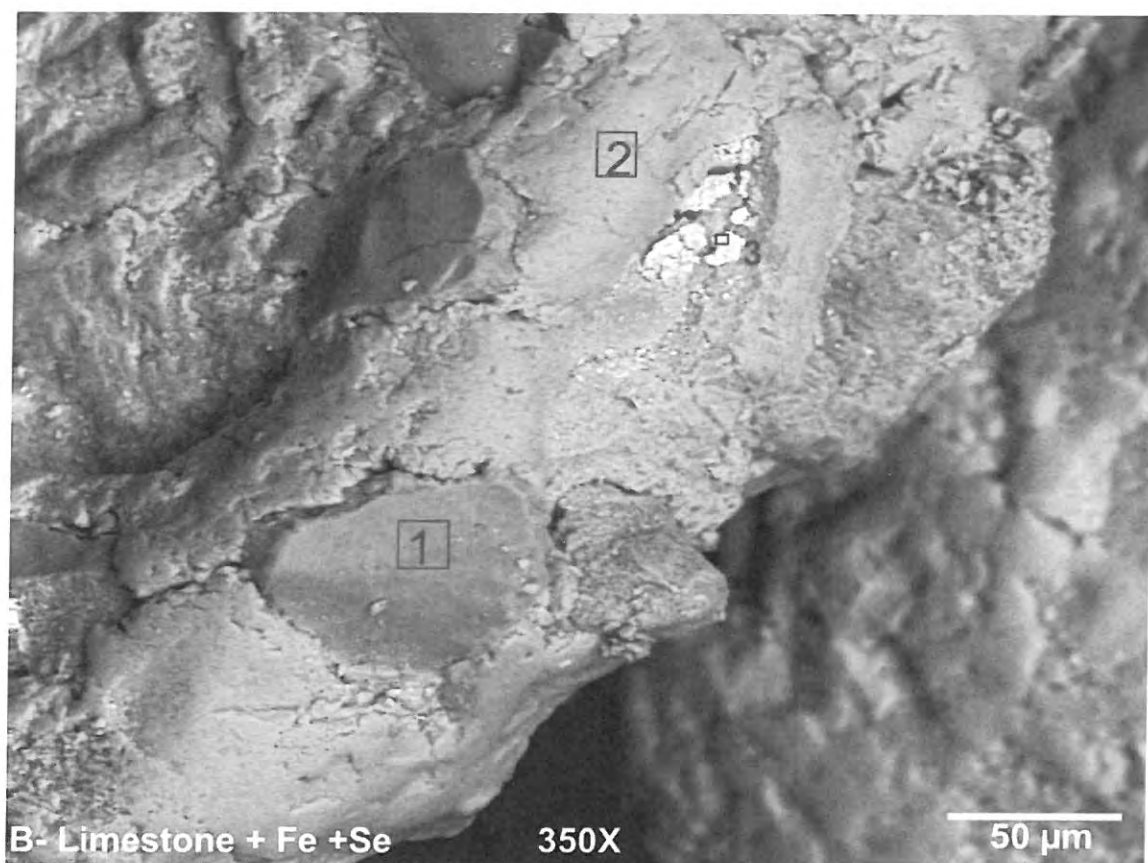
Figure 27. SEM image of the iron-coated limestone after the treatment with 100ppb

selenium solution

Analysis report:

Cursor=
Vert=991   Window 0.005 - 40.955=   41,194 cnt
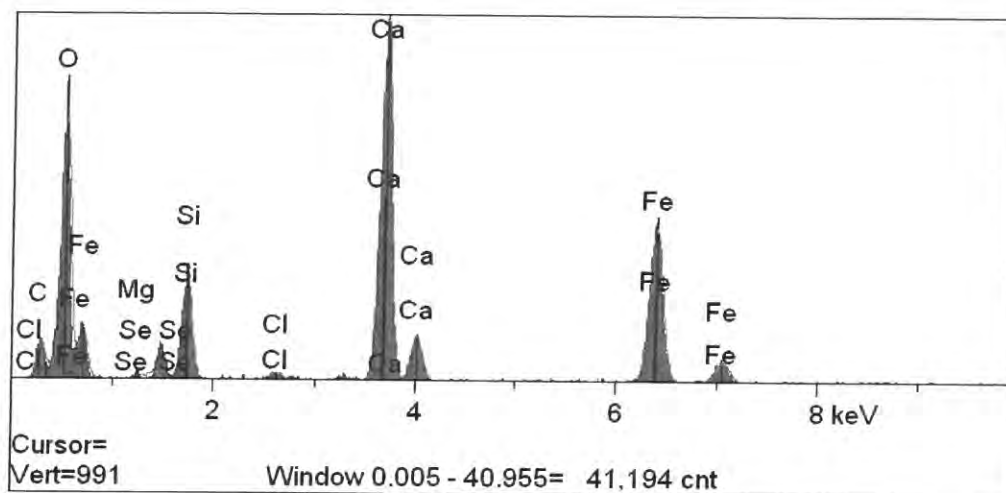
Table 20. SEM analysis of iron-coated limestone after the treatment with 100ppb

selenium solution

Elt.	Image-1		Image-2		Image-3	
	Atomic %	Conc wt%	Atomic %	Conc wt%	Atomic %	Conc wt%
C	0.00	0.00	16.24	10.18	11.43	6.55
O	68.18	53.34	66.75	55.70	69.04	52.73
Mg	0.00	0.00	0.72	0.91	0.12	0.14
Si	27.28	37.47	1.84	2.69	3.41	4.57
Cl	0.00	0.00	0.00	0.00	0.21	0.36
Ca	4.16	8.15	14.09	29.46	8.59	16.43
Fe	0.38	1.05	0.36	1.06	7.18	19.13
Se	0.00	0.00	0.00	0.00	0.02	0.07
Total	100.00	100.00	100.00	100.00	100.00	100.00

The analysis of SEM images of the iron-coated limestone after the treatment with 100ppb selenium solution indicates that the limestone has a very small amount of selenium on the surface associated with higher iron levels. No specific images of a selenium mineral can be observed. It is probably chemisorbed to iron hydroxide and is diffusely distributed with iron.

D.2. X-Ray Diffraction technique:

X-Ray diffraction provides information about the crystal structure, chemical composition and physical properties of materials. Uncoated and the iron-coated limestone is ground, homogenized and analyzed to determine their composition using XRD.
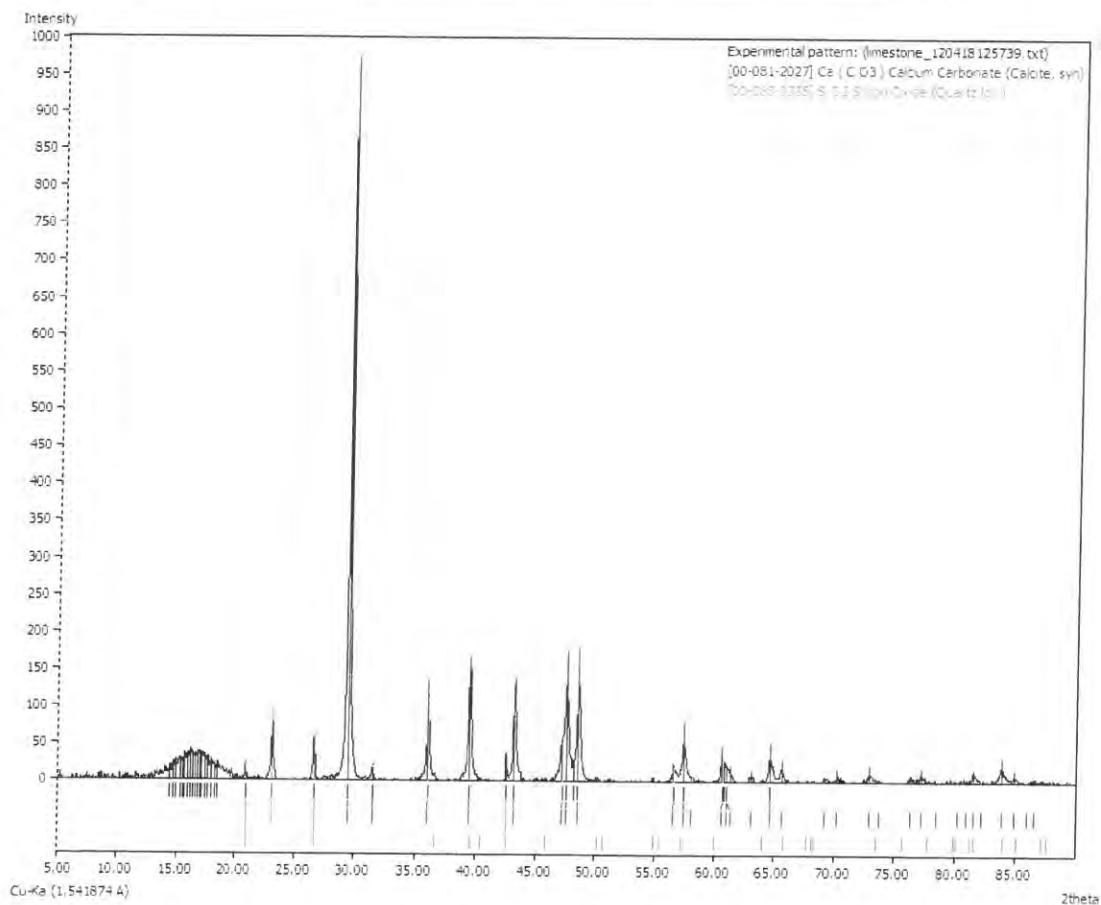
Figure 28. XRD pattern of uncoated limestone and $CaCO_3$

The XRD pattern of the uncoated limestone matches with the known reference
peaks of $CaCO_3$.
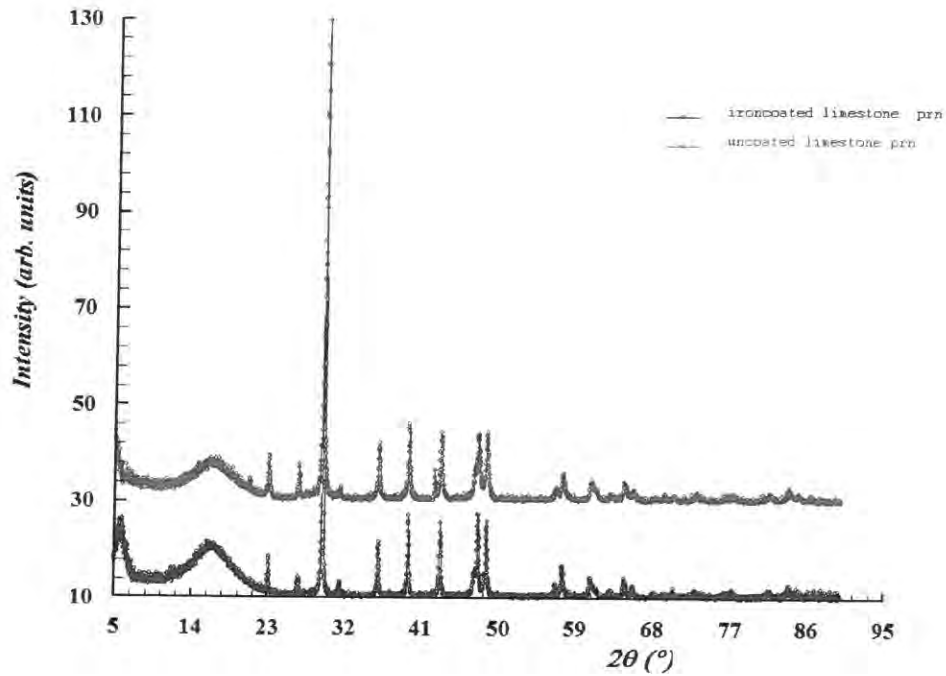
Figure 29. XRD pattern of uncoated and Iron-coated limestone

Little difference can be seen between the uncoated and iron-coated limestone.
Iron hydroxide is amorphous and finely distributed. It cannot be directly observed at this
level using XRD.

IV. CONCLUSIONS

Limestone is successfully used as a base material for the removal of the selected heavy metals. Plain limestone is coated with iron using iron (III) chloride solution and the efficiency of iron-coated and plain limestones were compared using batch and kinetic experiments. The effect of pH on the removal capacity of limestone was also studied.

The effect of contact time was studied in kinetics studies. It was found that 5 grams of the uncoated limestone removes the lead from a solution of 50ppb concentration within 10 minutes to below the drinking water standard. Over 99% of the lead from a 100mL solution of 100ppb concentration and a 100mL solution of 200ppb concentration was also removed quickly. A level of 5 grams iron-coated limestone removes lead completely from a solution of 50ppb concentration in 10 hours, however the drinking water standard was met within minutes. The uncoated limestone is more effective than the iron-coated limestone, because uncoated limestone removes lead completely in 30 minutes.

Selenium has also been quickly reduced to less than its drinking water standard. Within 5 hours, 99.4% of the selenium was removed after the treatment of 100mL solution of 50ppb concentration with 5grams of iron-coated limestone. It was also found that 92.5% of the selenium was removed in 4 hours after the treatment of 100mL solution of 100ppb concentration with 5 grams of iron-coated solution and 94.6% of the selenium was removed in 3 hours after the treatment of 100mL solution of 200ppb concentration with 5 grams of iron-coated solution. However, the drinking water standard was achieved

49

much sooner. Uncoated limestone removes 79.62% of the selenium from a 100mL solution of 50ppb concentration in 24 hours and was less effective than iron-coated limestone because it removes lead to below the drinking water standard in 30 minutes.

The effect of different amounts of uncoated limestone (5, 10, 20, 50 and 100g) to remove lead was studied. A sample of 10 grams of uncoated limestone efficiently removes lead completely from a 100mL solution of 50ppb. Additionally, 20 grams of uncoated limestone removes 99.8% of the lead from a 100mL solution of 100ppb and 50 grams of limestone efficiently removes 99.9% of lead from a 100mL solution of 200ppb concentration. However, drinking water standards were easily met with 5 grams of uncoated limestone.

Batch tests with limestone are very effective with all amounts. However, 20 grams of iron-coated limestone is most effective at removing selenium from a 100mL solution of 100ppb concentration, with 95.9% of selenium from the solution. Iron-coated limestone worked better for selenium because it removes selenium to below the drinking water standards in less than 4 hours.

The effect of pH on the efficiency of limestone to remove lead was studied. A pH of 6 is most efficient for removing lead from a 100mL solution of 100ppb. At pH 6, 73% of the lead has been removed. Results were widely varied over pH, however, which indicates the need for further study.

The effect of pH on the efficiency of iron-coated limestone to remove selenium was also studied. A pH 6 is more effective to remove selenium from a 100mL solution of

100ppb concentration. However, all other pH conditions tested were also effective to remove selenium below the drinking water standard.

Efficiencies of the uncoated and iron-coated limestones to remove heavy metals from drinking water were compared through batch and kinetic tests. Uncoated limestone was found to be more effective to remove lead. The mechanism for the removal of lead is probably precipitation as lead hydroxide. Iron-coated limestone was found to be more effective to remove selenium and is probably chemisorbed to iron hydroxide. This can be considered as chemisorption.

V. FUTURE WORK

Limestone is readily available and is relatively inexpensive for heavy metal removal. This technology can be adapted to small, rural water supply systems to reduce the heavy metals below their drinking water standards. Different time intervals can be used to study the effect on removal of metals. The effect of pH needs further studies. The mechanism for the removal of lead could be precipitation and needs to be studied further. selenium is removed by binding with iron through oxygen, this is considered as adsorption and further research need to be done to study this mechanism.

Different concentrations of iron (III) chloride can be used to coat the limestone and their effect to remove heavy metals can be compared. We can apply this method to remove combinations of metals.

VI. PERSPECTIVE

Drinking water is polluted with metals that are harmful for people due to waste from industries. There are ways to remove the metals from drinking water, but they are hard and costly. We came up with an idea to remove lead and selenium with a really simple cheap material. Our material is limestone. Limestone is available everywhere. Metals are attracted to the surface of the limestone and can be removed easily. Plain limestone worked very well for lead, but not for selenium. So, we just coated the limestone with a small amount of iron and this removed all the selenium. After the limestone is full, we can just take the limestone to a cement plant to be used in cement because the metals won't come off.

REFERENCES

1. Barry, R.P., Natalie, H. and Macintosh, D. Longitudinal Investigation of Exposure to Arsenic, Cadmium, and Lead in Drinking Water. *Environmental Health Perspectives*, **2000**, volume 108

2. Brookins, D.G. Eh-pH diagrams for geochemistry, Springer-Verlag New York, **1988**, 176p.

3. Campbell, B.C., Beattie, A.D., Moore, M.R., Goldburg, A. and Reid, A.G. Renal insufficiency associated with excessive lead exposure. *British medical journal*, **1977**, 1:482-485

4. Chappell, W.R., Beck, B., Brown, K.G., Chaney, R., Cothern, C.R., Irgolic, K.J., North, D.W., Thornton, I. and Tsongas, T.A. Inorganic arsenic: a need and an opportunity to improve risk assessment. *Environ Health Perspect* 105:1060-1067 (**1997**)

5. Dambies, L. Existing and prospective sorption technologies for the removal of arsenic in water. *Sep. Sci. Technol.* **2004**, *39*, 603-627

6. Galal, H.G. Dietary intake of pesticide residues, cadmium, mercury and lead. *Food additives and contaminants*, **1991**, 8(6):793-806

7. *Lead—environmental aspects* (Environmental Health Criteria, No. 85) Geneva, World Health Organization, **1989**

8. Lizama, A.K., Fletcher, T.D. and Sun, G. Enhancing the removal of arsenic, boron and heavy metals in subsurface flow constructed wetlands using different supporting media. *Water Science & Technology*. **2011**, 63(11), 2612-2618

9. Lobinski, R. Cytotoxic, Mutagenic and Carcinogenic Potential of Heavy Metals Related to Human Environment. *NATO ASI Ser., Ser. 2* **1997**, *26*, 389–409

10. Mark J.H. An assessment of current standards for selenium in drinking water. *Ground water.* **1981**, 19(4), 366-369

11. McNeill, L.S. and Edwards, M. Predicting arsenate removal during metal hydroxide precipitation. *J. Am. Water Works Assoc.* **1997**, *89* (1), 75-86

12. Mercedesdiaz, S. and M. Rosamartinez, T. Retention of Arsenic and Selenium Compounds Using Limestone in a Coal Gasification Flue Gas. *Environ. Sci. Technol.* **2004**, 38, 899-903

13. National Research Council. *Recommended dietary allowances*, 10th ed. Washington, DC, National Academy Press, **1989**

14. Pattanayak, J., Mondal, K., Mathew, S. and Lalvani, S.B. Aparametric evaluation of the removal of As(V) and As(III) by carbon-based adsorbents. *Carbon* **2000**, *38*, 589-596

15. Reed, B.E., Vaughan, R. and Jiang, L.Q. As(III), As(V), Hg, and Pb removal by Fe-oxide impregnated activated carbon. *J. Environ. Eng. Div. (Am. Soc. Civ. Eng.)* **2000**, *126*, 869-873

16. Schock, M.R. Understanding lead corrosion control strategies. *Journal of the American Water Works Association*, **1989**, 81:88

17. Selenium Concentrations in Natural and Environmental Waters, *Chem. Rev.* **1997**, 97, 1979-2003

18. *Selenium.* Geneva, World Health Organization, **1987** (Environmental Health Criteria, No.58

19. Sherlock, J.C., Ashby, D., Delves, H.T., Forbes, G.I., Moore, M.R., Patterson, W.J., Pocock, S.J., Quinn, M.J., Richards, W.N. and Wilson, T.S. Reduction in exposure to lead from drinking water and its effect on blood lead concentrations. *Human toxicology*, **1984**, 3:383-392

20. Sioris, L.J., Cuthrie, K. and Pentel, P.R. Acute selenium poisoning. *Veterinary and human toxicology*, **1980**, 22:364

21. Smith, M.J. and Westfall, B.B. Further field studies on the selenium problem in relation to public health. *US Public Health Report*, **1937**, 52:1375-1384

22. Waypa, J.J., Elimelech, M. and Hering, J.G. Removal of arsenic from water by membrane processes. *J. Am. Water Works Assoc.* **1997**, *89* (10), 102-114

23. Xiu, P.Y., Robert, K. and Jim, M.H. Determination of (Ultra)trace Amounts of Arsenic(III) and Arsenic(V) in Water by Inductively Coupled Plasma Mass Spectrometry Coupled with Flow Injection On-Line Sorption Preconcentration and Separation in a Knotted Reactor. *Anal. Chem.* **1998,** 70, 4736-4742

24. Zhimang, G., Jun, F. and Baolin, D. Preparation and Evaluation of GAC-Based Iron-Containing Adsorbents for Arsenic Removal. *Environ. Sci. Technol.* **2005**, 39, 3833-3843

ATTACHMENT 2

ANALYTICAL REPORT

Lab Number:	L1534367
Client:	Groundwater Treatment & Technology
	627 Mount Hope Road
	Wharton, NJ 07885
ATTN:	Rob Orlando
Phone:	(973) 983-0901
Project Name:	WURTSBORO MINE
Project Number:	Not Specified
Report Date:	12/31/15

Project Name:	WURTSBORO MINE	
Project Number:	Not Specified	

Lab Number:	L1534367
Report Date:	12/31/15

Alpha Sample ID	Client ID	Matrix	Sample Location	Collection Date/Time	Receive Date
L1534367-01	UNTREATED	WATER	WURTSBORO, NY	12/27/15 16:00	12/30/15
L1534367-02	3 LITER AA	WATER	WURTSBORO, NY	12/27/15 17:25	12/30/15
L1534367-03	8 LITER AA	WATER	WURTSBORO, NY	12/28/15 09:15	12/30/15
L1534367-04	13 LITER AA	WATER	WURTSBORO, NY	12/28/15 11:30	12/30/15
L1534367-05	21 LITER AA	WATER	WURTSBORO, NY	12/28/15 15:00	12/30/15
L1534367-06	39 LITER AA	WATER	WURTSBORO, NY	12/29/15 12:00	12/30/15
L1534367-07	3 LITER LIMESTONE	WATER	WURTSBORO, NY	12/27/15 17:36	12/30/15
L1534367-08	8 LITER LIMESTONE	WATER	WURTSBORO, NY	12/28/15 10:20	12/30/15
L1534367-09	11 LITER LIMESTONE	WATER	WURTSBORO, NY	12/28/15 13:15	12/30/15
L1534367-10	17 LITER LIMESTONE	WATER	WURTSBORO, NY	12/28/15 19:00	12/30/15
L1534367-11	35 LITER LIMESTONE	WATER	WURTSBORO, NY	12/29/15 12:00	12/30/15

Case Narrative

The samples were received in accordance with the Chain of Custody and no significant deviations were encountered during the preparation or analysis unless otherwise noted. Sample Receipt, Container Information, and the Chain of Custody are located at the back of the report.

Results contained within this report relate only to the samples submitted under this Alpha Lab Number and meet all of the requirements of NELAC, for all NELAC accredited parameters. The data presented in this report is organized by parameter (i.e. VOC, SVOC, etc.). Sample specific Quality Control data (i.e. Surrogate Spike Recovery) is reported at the end of the target analyte list for each individual sample, followed by the Laboratory Batch Quality Control at the end of each parameter. Tentatively Identified Compounds (TICs), if requested, are reported for compounds identified to be present and are not part of the method/program Target Compound List, even if only a subset of the TCL are being reported. If a sample was re-analyzed or re-extracted due to a required quality control corrective action and if both sets of data are reported, the Laboratory ID of the re-analysis or re-extraction is designated with an "R" or "RE", respectively. When multiple Batch Quality Control elements are reported (e.g. more than one LCS), the associated samples for each element are noted in the grey shaded header line of each data table. Any Laboratory Batch, Sample Specific % recovery or RPD value that is outside the listed Acceptance Criteria is bolded in the report. All specific QC information is also incorporated in the Data Usability format of our Data Merger tool where it can be reviewed along with any associated usability implications. Soil/sediments, solids and tissues are reported on a dry weight basis unless otherwise noted. Definitions of all data qualifiers and acronyms used in this report are provided in the Glossary located at the back of the report.

In reference to questions H (CAM) or 4 (RCP) when "NO" is checked, the performance criteria for CAM and RCP methods allow for some quality control failures to occur and still be within method compliance. In these instances the specific failure is not narrated but noted in the associated QC table. The information is also incorporated in the Data Usability format of our Data Merger tool where it can be reviewed along with any associated usability implications.

Please see the associated ADEx data file for a comparison of laboratory reporting limits that were achieved with the regulatory Numerical Standards requested on the Chain of Custody.

HOLD POLICY
For samples submitted on hold, Alpha's policy is to hold samples (with the exception of Air canisters) free of charge for 21 calendar days from the date the project is completed. After 21 calendar days, we will dispose of all samples submitted including those put on hold unless you have contacted your Client Service Representative and made arrangements for Alpha to continue to hold the samples. Air canisters will be disposed after 3 business days from the date the project is completed.

Please contact Client Services at 800-624-9220 with any questions.

| Project Name: | WURTSBORO MINE | Lab Number: | L1534367 |
| Project Number: | Not Specified | Report Date: | 12/31/15 |

Case Narrative (continued)

Report Submission

All non-detect (ND) or estimated concentrations (J-qualified) have been quantitated to the limit noted in the MDL column.

Sample Receipt

Unpreserved containers for pH were not received and the laboratory was not able to perform this requested analysis.

I, the undersigned, attest under the pains and penalties of perjury that, to the best of my knowledge and belief and based upon my personal inquiry of those responsible for providing the information contained in this analytical report, such information is accurate and complete. This certificate of analysis is not complete unless this page accompanies any and all pages of this report.

Authorized Signature: _Kelly Stenstrom_ Kelly Stenstrom

Title: Technical Director/Representative

Date: 12/31/15

ΔLPHA

METALS

| Project Name: | WURTSBORO MINE | | Lab Number: | L1534367 |
| Project Number: | Not Specified | | Report Date: | 12/31/15 |

SAMPLE RESULTS

Lab ID:	L1534367-01	Date Collected:	12/27/15 16:00
Client ID:	UNTREATED	Date Received:	12/30/15
Sample Location:	WURTSBORO, NY	Field Prep:	Not Specified
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Prep Method	Analytical Method	Analyst
Total Metals - Westborough Lab											
Lead, Total	0.5364		mg/l	0.00100	0.00025	2	12/30/15 12:50	12/31/15 11:36	EPA 3005A	1,6020A	KL

| Project Name: | WURTSBORO MINE | | Lab Number: | L1534367 |
| Project Number: | Not Specified | | Report Date: | 12/31/15 |

SAMPLE RESULTS

Lab ID:	L1534367-02	Date Collected:	12/27/15 17:25
Client ID:	3 LITER AA	Date Received:	12/30/15
Sample Location:	WURTSBORO, NY	Field Prep:	Not Specified
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Prep Method	Analytical Method	Analyst
Total Metals - Westborough Lab											
Lead, Total	0.00918		mg/l	0.00050	0.00012	1	12/30/15 12:50	12/31/15 10:37	EPA 3005A	1,6020A	KL

| **Project Name:** | WURTSBORO MINE | | **Lab Number:** | L1534367 |
| **Project Number:** | Not Specified | | **Report Date:** | 12/31/15 |

SAMPLE RESULTS

Lab ID:	L1534367-03	**Date Collected:**	12/28/15 09:15
Client ID:	8 LITER AA	**Date Received:**	12/30/15
Sample Location:	WURTSBORO, NY	**Field Prep:**	Not Specified
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Prep Method	Analytical Method	Analyst
Total Metals - Westborough Lab											
Lead, Total	0.04024		mg/l	0.00050	0.00012	1	12/30/15 12:50	12/31/15 10:41	EPA 3005A	1,6020A	KL

| Project Name: | WURTSBORO MINE | | Lab Number: | L1534367 |
| Project Number: | Not Specified | | Report Date: | 12/31/15 |

SAMPLE RESULTS

Lab ID:	L1534367-04	Date Collected:	12/28/15 11:30
Client ID:	13 LITER AA	Date Received:	12/30/15
Sample Location:	WURTSBORO, NY	Field Prep:	Not Specified
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Prep Method	Analytical Method	Analyst
Total Metals - Westborough Lab											
Lead, Total	0.04378		mg/l	0.00050	0.00012	1	12/30/15 12:50	12/31/15 10:11	EPA 3005A	1,6020A	KL

ΔLPHA

| Project Name: | WURTSBORO MINE | | Lab Number: | L1534367 |
| Project Number: | Not Specified | | Report Date: | 12/31/15 |

SAMPLE RESULTS

Lab ID:	L1534367-05	Date Collected:	12/28/15 15:00
Client ID:	21 LITER AA	Date Received:	12/30/15
Sample Location:	WURTSBORO, NY	Field Prep:	Not Specified
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Prep Method	Analytical Method	Analyst
Total Metals - Westborough Lab											
Lead, Total	0.04868		mg/l	0.00050	0.00012	1	12/30/15 12:50	12/31/15 10:44	EPA 3005A	1,6020A	KL

ALPHA

| Project Name: | WURTSBORO MINE | | Lab Number: | L1534367 |
| Project Number: | Not Specified | | Report Date: | 12/31/15 |

SAMPLE RESULTS

Lab ID:	L1534367-06	Date Collected:	12/29/15 12:00
Client ID:	39 LITER AA	Date Received:	12/30/15
Sample Location:	WURTSBORO, NY	Field Prep:	Not Specified
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Prep Method	Analytical Method	Analyst
Total Metals - Westborough Lab											
Lead, Total	0.09458		mg/l	0.00050	0.00012	1	12/30/15 12:50	12/31/15 10:48	EPA 3005A	1,6020A	KL

ᐃLPHA

| **Project Name:** | WURTSBORO MINE | | **Lab Number:** | L1534367 |
| **Project Number:** | Not Specified | | **Report Date:** | 12/31/15 |

SAMPLE RESULTS

Lab ID:	L1534367-07	**Date Collected:**	12/27/15 17:36
Client ID:	3 LITER LIMESTONE	**Date Received:**	12/30/15
Sample Location:	WURTSBORO, NY	**Field Prep:**	Not Specified
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Prep Method	Analytical Method	Analyst
Total Metals - Westborough Lab											
Lead, Total	0.00045	J	mg/l	0.00050	0.00012	1	12/30/15 12:50	12/31/15 10:52	EPA 3005A	1,6020A	KL

Project Name:	WURTSBORO MINE		**Lab Number:**	L1534367
Project Number:	Not Specified		**Report Date:**	12/31/15

SAMPLE RESULTS

Lab ID:	L1534367-08		Date Collected:	12/28/15 10:20
Client ID:	8 LITER LIMESTONE		Date Received:	12/30/15
Sample Location:	WURTSBORO, NY		Field Prep:	Not Specified
Matrix:	Water			

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Prep Method	Analytical Method	Analyst
Total Metals - Westborough Lab											
Lead, Total	0.00039	J	mg/l	0.00050	0.00012	1	12/30/15 12:50	12/31/15 10:55	EPA 3005A	1,6020A	KL

| **Project Name:** | WURTSBORO MINE | | **Lab Number:** | L1534367 |
| **Project Number:** | Not Specified | | **Report Date:** | 12/31/15 |

SAMPLE RESULTS

Lab ID:	L1534367-09	**Date Collected:**	12/28/15 13:15
Client ID:	11 LITER LIMESTONE	**Date Received:**	12/30/15
Sample Location:	WURTSBORO, NY	**Field Prep:**	Not Specified
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Prep Method	Analytical Method	Analyst
Total Metals - Westborough Lab											
Lead, Total	0.00158		mg/l	0.00050	0.00012	1	12/30/15 12:50	12/31/15 10:59	EPA 3005A	1,6020A	KL

| Project Name: | WURTSBORO MINE | | Lab Number: | L1534367 |
| Project Number: | Not Specified | | Report Date: | 12/31/15 |

SAMPLE RESULTS

Lab ID:	L1534367-10	Date Collected:	12/28/15 19:00
Client ID:	17 LITER LIMESTONE	Date Received:	12/30/15
Sample Location:	WURTSBORO, NY	Field Prep:	Not Specified
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Prep Method	Analytical Method	Analyst
Total Metals - Westborough Lab											
Lead, Total	0.00045	J	mg/l	0.00050	0.00012	1	12/30/15 12:50	12/31/15 11:03	EPA 3005A	1,6020A	KL

| Project Name: | WURTSBORO MINE | | Lab Number: | L1534367 |
| Project Number: | Not Specified | | Report Date: | 12/31/15 |

SAMPLE RESULTS

Lab ID:	L1534367-11	Date Collected:	12/29/15 12:00
Client ID:	35 LITER LIMESTONE	Date Received:	12/30/15
Sample Location:	WURTSBORO, NY	Field Prep:	Not Specified
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Prep Method	Analytical Method	Analyst
Total Metals - Westborough Lab											
Lead, Total	0.00048	J	mg/l	0.00050	0.00012	1	12/30/15 12:50	12/31/15 11:06	EPA 3005A	1,6020A	KL

ALPHA

| Project Name: | WURTSBORO MINE | Lab Number: | L1534367 |
| Project Number: | Not Specified | Report Date: | 12/31/15 |

Method Blank Analysis
Batch Quality Control

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
Total Metals - Westborough Lab for sample(s): 01-11 Batch: WG853962-1										
Lead, Total	ND		mg/l	0.00050	0.00012	1	12/30/15 12:50	12/31/15 10:00	1,6020A	KL

Prep Information

Digestion Method: EPA 3005A

Lab Control Sample Analysis
Batch Quality Control

Project Name:	WURTSBORO MINE
Project Number:	Not Specified

Lab Number:	L1534367
Report Date:	12/31/15

Parameter	LCS %Recovery	Qual	LCSD %Recovery	Qual	%Recovery Limits	RPD	Qual	RPD Limits
Total Metals - Westborough Lab Associated sample(s): 01-11 Batch: WG853962-2								
Lead, Total	104		-		80-120	-		

ALPHA

Matrix Spike Analysis
Batch Quality Control

Project Name: WURTSBORO MINE

Project Number: Not Specified

Lab Number: L1534367

Report Date: 12/31/15

Parameter	Native Sample	MS Added	MS Found	MS %Recovery	Qual	MSD Found	MSD %Recovery	Qual	Recovery Limits	RPD	Qual	RPD Limits
Total Metals - Westborough Lab Associated sample(s): 01-11 QC Batch ID: WG853962-4 QC Sample: L1534367-04 Client ID: 13 LITER AA												
Lead, Total	0.04378	0.51	0.5784	105		-	-		75-125	-		20

ALPHA

Lab Duplicate Analysis
Batch Quality Control

| Project Name: | WURTSBORO MINE | | Lab Number: | L1534367 |
| Project Number: | Not Specified | | Report Date: | 12/31/15 |

Parameter	Native Sample	Duplicate Sample	Units	RPD	Qual	RPD Limits
Total Metals - Westborough Lab Associated sample(s): 01-11 QC Batch ID: WG853962-3 QC Sample: L1534367-04 Client ID: 13 LITER AA						
Lead, Total	0.04378	0.04332	mg/l	1		20

ALPHA

Project Name: WURTSBORO MINE
Project Number: Not Specified

Sample Receipt and Container Information

Were project specific reporting limits specified?　　　YES

Cooler Information Custody Seal
Cooler
A　　　　　　　　Absent

Container Information

Container ID	Container Type	Cooler	pH	Temp deg C	Pres	Seal	Analysis(*)
L1534367-01A	Plastic 250ml HNO3 preserved	A	<2	5.7	Y	Absent	PB-6020T(180)
L1534367-02A	Plastic 250ml HNO3 preserved	A	<2	5.7	Y	Absent	PB-6020T(180)
L1534367-03A	Plastic 250ml HNO3 preserved	A	<2	5.7	Y	Absent	PB-6020T(180)
L1534367-04A	Plastic 250ml HNO3 preserved	A	<2	5.7	Y	Absent	PB-6020T(180)
L1534367-05A	Plastic 250ml HNO3 preserved	A	<2	5.7	Y	Absent	PB-6020T(180)
L1534367-06A	Plastic 250ml HNO3 preserved	A	<2	5.7	Y	Absent	PB-6020T(180)
L1534367-07A	Plastic 250ml HNO3 preserved	A	<2	5.7	Y	Absent	PB-6020T(180)
L1534367-08A	Plastic 250ml HNO3 preserved	A	<2	5.7	Y	Absent	PB-6020T(180)
L1534367-09A	Plastic 250ml HNO3 preserved	A	<2	5.7	Y	Absent	PB-6020T(180)
L1534367-10A	Plastic 250ml HNO3 preserved	A	<2	5.7	Y	Absent	PB-6020T(180)
L1534367-11A	Plastic 250ml HNO3 preserved	A	<2	5.7	Y	Absent	PB-6020T(180)

*Values in parentheses indicate holding time in days

ALPHA

Project Name:	WURTSBORO MINE	**Lab Number:**	L1534367
Project Number:	Not Specified	**Report Date:**	12/31/15

GLOSSARY

Acronyms

EDL	- Estimated Detection Limit: This value represents the level to which target analyte concentrations are reported as estimated values, when those target analyte concentrations are quantified below the reporting limit (RL). The EDL includes any adjustments from dilutions, concentrations or moisture content, where applicable. The use of EDLs is specific to the analysis of PAHs using Solid-Phase Microextraction (SPME).
EPA	- Environmental Protection Agency.
LCS	- Laboratory Control Sample: A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes.
LCSD	- Laboratory Control Sample Duplicate: Refer to LCS.
LFB	- Laboratory Fortified Blank: A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes.
MDL	- Method Detection Limit: This value represents the level to which target analyte concentrations are reported as estimated values, when those target analyte concentrations are quantified below the reporting limit (RL). The MDL includes any adjustments from dilutions, concentrations or moisture content, where applicable.
MS	- Matrix Spike Sample: A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.
MSD	- Matrix Spike Sample Duplicate: Refer to MS.
NA	- Not Applicable.
NC	- Not Calculated: Term is utilized when one or more of the results utilized in the calculation are non-detect at the parameter's reporting unit.
NI	- Not Ignitable.
NP	- Non-Plastic: Term is utilized for the analysis of Atterberg Limits in soil.
RL	- Reporting Limit: The value at which an instrument can accurately measure an analyte at a specific concentration. The RL includes any adjustments from dilutions, concentrations or moisture content, where applicable.
RPD	- Relative Percent Difference: The results from matrix and/or matrix spike duplicates are primarily designed to assess the precision of analytical results in a given matrix and are expressed as relative percent difference (RPD). Values which are less than five times the reporting limit for any individual parameter are evaluated by utilizing the absolute difference between the values; although the RPD value will be provided in the report.
SRM	- Standard Reference Material: A reference sample of a known or certified value that is of the same or similar matrix as the associated field samples.
STLP	- Semi-dynamic Tank Leaching Procedure per EPA Method 1315.
TIC	- Tentatively Identified Compound: A compound that has been identified to be present and is not part of the target compound list (TCL) for the method and/or program. All TICs are qualitatively identified and reported as estimated concentrations.

Footnotes

1	- The reference for this analyte should be considered modified since this analyte is absent from the target analyte list of the original method.

Terms

Total: With respect to Organic analyses, a 'Total' result is defined as the summation of results for individual isomers or Aroclors. If a 'Total' result is requested, the results of its individual components will also be reported. This is applicable to 'Total' results for methods 8260, 8081 and 8082.

Analytical Method: Both the document from which the method originates and the analytical reference method. (Example: EPA 8260B is shown as 1,8260B.) The codes for the reference method documents are provided in the References section of the Addendum.

Data Qualifiers

A	- Spectra identified as "Aldol Condensation Product".
B	- The analyte was detected above the reporting limit in the associated method blank. Flag only applies to associated field samples that have detectable concentrations of the analyte at less than ten times (10x) the concentration found in the blank. For MCP-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte at less than ten times (10x) the concentration found in the blank. For DOD-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte at less than ten times (10x) the concentration found in the blank AND the analyte was detected above one-half the reporting limit (or above the reporting limit for common lab contaminants) in the associated method blank. For NJ-Air-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte above the reporting limit. For NJ-related projects (excluding Air), flag only applies to associated field samples that have detectable concentrations of the analyte, which was detected above the reporting limit in the associated method blank or above five times the reporting limit for common lab contaminants (Phthalates, Acetone, Methylene Chloride, 2-Butanone).

Report Format: DU Report with 'J' Qualifiers

Project Name:	WURTSBORO MINE	Lab Number:	L1534367
Project Number:	Not Specified	Report Date:	12/31/15

Data Qualifiers

C   - Co-elution: The target analyte co-elutes with a known lab standard (i.e. surrogate, internal standards, etc.) for co-extracted analyses.

D   - Concentration of analyte was quantified from diluted analysis. Flag only applies to field samples that have detectable concentrations of the analyte.

E   - Concentration of analyte exceeds the range of the calibration curve and/or linear range of the instrument.

G   - The concentration may be biased high due to matrix interferences (i.e. co-elution) with non-target compound(s). The result should be considered estimated.

H   - The analysis of pH was performed beyond the regulatory-required holding time of 15 minutes from the time of sample collection.

I   - The lower value for the two columns has been reported due to obvious interference.

M   - Reporting Limit (RL) exceeds the MCP CAM Reporting Limit for this analyte.

NJ   - Presumptive evidence of compound. This represents an estimated concentration for Tentatively Identified Compounds (TICs), where the identification is based on a mass spectral library search.

P   - The RPD between the results for the two columns exceeds the method-specified criteria.

Q   - The quality control sample exceeds the associated acceptance criteria. For DOD-related projects, LCS and/or Continuing Calibration Standard exceedences are also qualified on all associated sample results. Note: This flag is not applicable for matrix spike recoveries when the sample concentration is greater than 4x the spike added or for batch duplicate RPD when the sample concentrations are less than 5x the RL. (Metals only.)

R   - Analytical results are from sample re-analysis.

RE   - Analytical results are from sample re-extraction.

S   - Analytical results are from modified screening analysis.

J   - Estimated value. The Target analyte concentration is below the quantitation limit (RL), but above the Method Detection Limit (MDL) or Estimated Detection Limit (EDL) for SPME-related analyses. This represents an estimated concentration for Tentatively Identified Compounds (TICs).

ND   - Not detected at the method detection limit (MDL) for the sample, or estimated detection limit (EDL) for SPME-related analyses.

Report Format: DU Report with 'J' Qualifiers

ALPHA

REFERENCES

1   Test Methods for Evaluating Solid Waste: Physical/Chemical Methods. EPA SW-846.
      Third Edition. Updates I - IV, 2007.

LIMITATION OF LIABILITIES

Alpha Analytical performs services with reasonable care and diligence normal to the analytical testing laboratory industry. In the event of an error, the sole and exclusive responsibility of Alpha Analytical shall be to re-perform the work at it's own expense. In no event shall Alpha Analytical be held liable for any incidental, consequential or special damages, including but not limited to, damages in any way connected with the use of, interpretation of, information or analysis provided by Alpha Analytical.

We strongly urge our clients to comply with EPA protocol regarding sample volume, preservation, cooling, containers, sampling procedures, holding time and splitting of samples in the field.

Alpha Analytical, Inc.
Facility: **Company-wide**
Department: **Quality Assurance**
Title: **Certificate/Approval Program Summary**

Certification Information

The following analytes are not included in our Primary NELAP Scope of Accreditation:

Westborough Facility
EPA 524.2: 1,2-Dibromo-3-chloropropane, 1,2-Dibromoethane, m/p-xylene, o-xylene
EPA 624: 2-Butanone (MEK), 1,4-Dioxane, tert-Amylmethyl Ether, tert-Butyl Alcohol, m/p-xylene, o-xylene
EPA 625: Aniline, Benzoic Acid, Benzyl Alcohol, 4-Chloroaniline, 3-Methylphenol, 4-Methylphenol.
EPA 1010A: NPW: Ignitability
EPA 6010C: NPW: Strontium; SCM: Strontium
EPA 8151A: NPW: 2,4-DB, Dicamba, Dichloroprop, MCPA, MCPP; SCM: 2,4-DB, Dichloroprop, MCPA, MCPP
EPA 8260C: NPW: 1,2,4,5-Tetramethylbenzene; 4-Ethyltoluene, Azobenzene, Isopropanol; SCM: Iodomethane (methyl iodide), Methyl methacrylate (soil); 1,2,4,5-Tetramethylbenzene; 4-Ethyltoluene.
EPA 8270D: NPW: Pentachloronitrobenzene, 1-Methylnaphthalene, Dimethylnaphthalene,1,4-Diphenylhydrazine; SCM: Pentachloronitrobenzene, 1-Methylnaphthalene, Dimethylnaphthalene,1,4-Diphenylhydrazine.
EPA 9010: NPW: Amenable Cyanide Distillation, Total Cyanide Distillation
EPA 9038: NPW: Sulfate
EPA 9050A: NPW: Specific Conductance
EPA 9056: NPW: Chloride, Nitrate, Sulfate
EPA 9065: NPW: Phenols
EPA 9251: NPW: Chloride
SM3500: NPW: Ferrous Iron
SM4500: NPW: Amenable Cyanide, Dissolved Oxygen; SCM: Total Phosphorus, TKN, NO2, NO3.
SM5310C: DW: Dissolved Organic Carbon

Mansfield Facility
EPA 8270D: NPW: Biphenyl; SCM: Biphenyl
EPA 2540D: TSS
EPA TO-15: Halothane, 2,4,4-Trimethyl-2-pentene, 2,4,4-Trimethyl-1-pentene, Thiophene, 2-Methylthiophene, 3-Methylthiophene, 2-Ethylthiophene, 1,2,3-Trimethylbenzene, Indan, Indene, 1,2,4,5-Tetramethylbenzene, Benzothiophene, 1-Methylnaphthalene.

The following analytes are included in our Massachusetts DEP Scope of Accreditation, Westborough Facility:

Drinking Water
EPA 200.8: Sb,As,Ba,Be,Cd,Cr,Cu,Pb,Ni,Se,Tl; **EPA 200.7:** Ba,Be,Ca,Cd,Cr,Cu,Na; **EPA 245.1:** Mercury;
EPA 300.0: Nitrate-N, Fluoride, Sulfate; **EPA 353.2:** Nitrate-N, Nitrite-N; **SM4500NO3-F:** Nitrate-N, Nitrite-N; **SM4500F-C, SM4500CN-CE, EPA 180.1, SM2130B, SM4500Cl-D, SM2320B, SM2540C, SM4500H-B**
EPA 332: Perchlorate.
Microbiology: SM9215B; SM9223-P/A, SM9223B-Colilert-QT, Enterolert-QT.

Non-Potable Water
EPA 200.8: Al,Sb,As,Be,Cd,Cr,Cu,Pb,Mn,Ni,Se,Ag,Tl,Zn;
EPA 200.7: Al,Sb,As,Be,Cd,Ca,Cr,Co,Cu,Fe,Pb,Mg,Mn,Mo,Ni,K,Se,Ag,Na,Sr,Ti,Tl,V,Zn;
EPA 245.1, SM4500H,B, EPA 120.1, SM2510B, SM2540C, SM2340B, SM2320B, SM4500CL-E, SM4500F-BC, SM426C, SM4500NH3-BH, EPA 350.1: Ammonia-N, **LACHAT 10-107-06-1-B:** Ammonia-N, **SM4500NO3-F,**
EPA 353.2: Nitrate-N, **SM4500NH3-BC-NES, EPA 351.1, SM4500P-E, SM4500P-B, E, SM5220D, EPA 410.4, SM5210B, SM5310C, SM4500CL-D, EPA 1664, SM14 510AC, EPA 420.1, SM4500-CN-CE, SM2540D.**
EPA 624: Volatile Halocarbons & Aromatics,
EPA 608: Chlordane, Toxaphene, Aldrin, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, Dieldrin, DDD, DDE, DDT, Endosulfan I, Endosulfan II, Endosulfan sulfate, Endrin, Endrin Aldehyde, Heptachlor, Heptachlor Epoxide, PCBs
EPA 625: SVOC (Acid/Base/Neutral Extractables), **EPA 600/4-81-045:** PCB-Oil.
Microbiology: SM9223B-Colilert-QT; Enterolert-QT, SM9222D-MF.

For a complete listing of analytes and methods, please contact your Alpha Project Manager.

Document Type: Form

Pre-Qualtrax Document ID: 08-113

CHAIN OF CUSTODY

PAGE _1_ OF _1_

ALPHA
WESTBORO, MA — TEL: 508-898-9220 — FAX: 508-898-9193
MANSFIELD, MA — TEL: 508-822-9300 — FAX: 508-822-3288

Date Rec'd in Lab: 12/30/15

ALPHA Job #: U534367

Project Information

Project Name: WURTSBORO MINE

Project Location: WURTSBORO NY

Project #:

Project Manager: ROBERT KUNZEL

ALPHA Quote #:

Client Information

Client: GROUND/WATER TREAT § TECH

Address: 627 MT HOPE RD
WHARTON NJ 07885

Phone: 973 983 0901

Fax: 973 983 0903

Email: RKUNZEL@GWTTLLC.COM

☐ These samples have been previously analyzed by Alpha

Other Project Specific Requirements/Comments/Detection Limits:
DETECTION LIMIT 1 PPb or LESS

Report Information - Data Deliverables

☐ FAX ☒ EMAIL
☐ ADEx ☐ Add'l Deliverables

Regulatory Requirements/Report Limits

State /Fed Program   Criteria

Billing Information

☒ Same as Client info   PO #: 09 RGK

Turn-Around Time

☐ Standard   ☐ RUSH (only confirmed if pre-approved)

Date Due: 1/4/16   Time: 2PM

ANALYSIS: LEAD, PH

SAMPLE HANDLING
Filtration_____
☐ Done
☒ Not needed
☐ Lab to do
Preservation
☐ Lab to do
(Please specify below)

TOTAL # BOTTLES

ALPHA Lab ID (Lab Use Only)	Sample ID	Collection Date	Collection Time	Sample Matrix	Sampler's Initials	Lead	PH							Sample Specific Comments
34367 -01	UNTREATED	12/27/15	16:00	W	RGK	X	X							
-02	3 LITER AA	12/27/15	17:25	W	RGK	X	X							
-03	8 LITER AA	12/28/15	9:15	W	RGK	X	X							
-04	13 LITER AA	12/22/15	11:30	W	RGK	X	X							
-05	21 LITER AA	12/28/15	15:00	W	RGK	X	X							
-06	39 LITER AA	12/29/15	12:00	W	RGK	X	X							
-07	3. LITER LIMESTONE	12/27/15	17:36	W	RGK	X	X							
-08	8 LITER "	12/28/15	10:20	W	RGK	X	X							
-09	11 LITER "	12/22/15	13:15	W	RGK	X	X							
-10	17 LITER "	12/22/15	19:00	W	RGK	X	X							
-19	35 LITER "	12/29/15	12:00	W	RGK	X	X							

Container Type: PLASTIC
Preservative: NITRIC

Relinquished By:	Date/Time	Received By:	Date/Time
[signature] KUNZEL	12/29/15 4 PM	[signature]	12/30/15 935

Please print clearly, legibly and completely. Samples can not be logged in and turnaround time clock will not start until any ambiguities are resolved. All samples submitted are subject to Alpha's Terms and Conditions. See reverse side.

FORM NO. 01-01 (rev. 14-OCT-07)

ATTACHMENT 3

ANALYTICAL REPORT

Lab Number:	L1600382
Client:	Groundwater Treatment & Technology
	627 Mount Hope Road
	Wharton, NJ 07885
ATTN:	Rob Orlando
Phone:	(973) 983-0901
Project Name:	WURTSBORO MINE
Project Number:	WURTSBORO MINE
Report Date:	01/11/16

Eight Walkup Drive, Westborough, MA 01581-1019
508-898-9220 (Fax) 508-898-9193 800-624-9220 - www.alphalab.com

Project Name: WURTSBORO MINE

Project Number: WURTSBORO MINE

Lab Number: L1600382

Report Date: 01/11/16

Alpha Sample ID	Client ID	Matrix	Sample Location	Collection Date/Time	Receive Date
L1600382-01	UNTREATED 2	WATER	WURTSBORO, NY	01/05/16 10:00	01/06/16
L1600382-02	UNTREATED 2	WATER	WURTSBORO, NY	01/05/16 10:00	01/06/16
L1600382-03	3 LITERS 3/8" LIMESTONE	WATER	WURTSBORO, NY	01/05/16 10:35	01/06/16
L1600382-04	8 LITERS 3/8" LIMESTONE	WATER	WURTSBORO, NY	01/05/16 11:25	01/06/16
L1600382-05	21 LITERS 3/8" LIMESTONE	WATER	WURTSBORO, NY	01/05/16 13:30	01/06/16
L1600382-06	39 LITERS 3/8" LIMESTONE	WATER	WURTSBORO, NY	01/05/16 16:05	01/06/16
L1600382-07	70 LITERS 3/8" LIMESTONE	WATER	WURTSBORO, NY	01/06/16 10:00	01/06/16
L1600382-08	70 LITERS 3/8" LIMESTONE	WATER	WURTSBORO, NY	01/06/16 10:00	01/06/16

Project Name: WURTSBORO MINE

Project Number: WURTSBORO MINE

Lab Number: L1600382

Report Date: 01/11/16

Case Narrative

The samples were received in accordance with the Chain of Custody and no significant deviations were encountered during the preparation or analysis unless otherwise noted. Sample Receipt, Container Information, and the Chain of Custody are located at the back of the report.

Results contained within this report relate only to the samples submitted under this Alpha Lab Number and meet all of the requirements of NELAC, for all NELAC accredited parameters. The data presented in this report is organized by parameter (i.e. VOC, SVOC, etc.). Sample specific Quality Control data (i.e. Surrogate Spike Recovery) is reported at the end of the target analyte list for each individual sample, followed by the Laboratory Batch Quality Control at the end of each parameter. Tentatively Identified Compounds (TICs), if requested, are reported for compounds identified to be present and are not part of the method/program Target Compound List, even if only a subset of the TCL are being reported. If a sample was re-analyzed or re-extracted due to a required quality control corrective action and if both sets of data are reported, the Laboratory ID of the re-analysis or re-extraction is designated with an "R" or "RE", respectively. When multiple Batch Quality Control elements are reported (e.g. more than one LCS), the associated samples for each element are noted in the grey shaded header line of each data table. Any Laboratory Batch, Sample Specific % recovery or RPD value that is outside the listed Acceptance Criteria is bolded in the report. All specific QC information is also incorporated in the Data Usability format of our Data Merger tool where it can be reviewed along with any associated usability implications. Soil/sediments, solids and tissues are reported on a dry weight basis unless otherwise noted. Definitions of all data qualifiers and acronyms used in this report are provided in the Glossary located at the back of the report.

In reference to questions H (CAM) or 4 (RCP) when "NO" is checked, the performance criteria for CAM and RCP methods allow for some quality control failures to occur and still be within method compliance. In these instances the specific failure is not narrated but noted in the associated QC table. The information is also incorporated in the Data Usability format of our Data Merger tool where it can be reviewed along with any associated usability implications.

Please see the associated ADEx data file for a comparison of laboratory reporting limits that were achieved with the regulatory Numerical Standards requested on the Chain of Custody.

HOLD POLICY

For samples submitted on hold, Alpha's policy is to hold samples (with the exception of Air canisters) free of charge for 21 calendar days from the date the project is completed. After 21 calendar days, we will dispose of all samples submitted including those put on hold unless you have contacted your Client Service Representative and made arrangements for Alpha to continue to hold the samples. Air canisters will be disposed after 3 business days from the date the project is completed.

Please contact Client Services at 800-624-9220 with any questions.

ΔLPHA

Project Name: WURTSBORO MINE

Project Number: WURTSBORO MINE

Lab Number: L1600382

Report Date: 01/11/16

Case Narrative (continued)

Report Submission

All non-detect (ND) or estimated concentrations (J-qualified) have been quantitated to the limit noted in the MDL column.

ALPHA
ANALYTICAL

METALS

| Project Name: | WURTSBORO MINE | | Lab Number: | L1600382 |
| Project Number: | WURTSBORO MINE | | Report Date: | 01/11/16 |

SAMPLE RESULTS

Lab ID:	L1600382-01	Date Collected:	01/05/16 10:00
Client ID:	UNTREATED 2	Date Received:	01/06/16
Sample Location:	WURTSBORO, NY	Field Prep:	Not Specified
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Prep Method	Analytical Method	Analyst
Total Metals - Westborough Lab											
Lead, Total	0.5172		mg/l	0.01000	0.00129	10	01/07/16 09:20	01/07/16 15:44	EPA 3005A	1,6020A	KL

ALPHA
ANALYTICAL

| Project Name: | WURTSBORO MINE | | Lab Number: | L1600382 |
| Project Number: | WURTSBORO MINE | | Report Date: | 01/11/16 |

SAMPLE RESULTS

Lab ID:	L1600382-02	Date Collected:	01/05/16 10:00
Client ID:	UNTREATED 2	Date Received:	01/06/16
Sample Location:	WURTSBORO, NY	Field Prep:	Not Specified
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Prep Method	Analytical Method	Analyst
Dissolved Metals - Westborough Lab											
Lead, Dissolved	0.4754		mg/l	0.00100	0.00012	1	01/07/16 12:05	01/07/16 15:55	EPA 3005A	1,6020A	KL

| Project Name: | WURTSBORO MINE | | Lab Number: | L1600382 |
| Project Number: | WURTSBORO MINE | | Report Date: | 01/11/16 |

SAMPLE RESULTS

Lab ID:	L1600382-03	Date Collected:	01/05/16 10:35
Client ID:	3 LITERS 3/8" LIMESTONE	Date Received:	01/06/16
Sample Location:	WURTSBORO, NY	Field Prep:	Not Specified
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Prep Method	Analytical Method	Analyst
Total Metals - Westborough Lab											
Lead, Total	0.2319		mg/l	0.00100	0.00012	1	01/07/16 09:20	01/07/16 14:10	EPA 3005A	1,6020A	KL

ALPHA
ANALYTICAL

Project Name:	WURTSBORO MINE		Lab Number:	L1600382
Project Number:	WURTSBORO MINE		Report Date:	01/11/16

SAMPLE RESULTS

Lab ID:	L1600382-04	Date Collected:	01/05/16 11:25
Client ID:	8 LITERS 3/8" LIMESTONE	Date Received:	01/06/16
Sample Location:	WURTSBORO, NY	Field Prep:	Not Specified
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Prep Method	Analytical Method	Analyst
Total Metals - Westborough Lab											
Lead, Total	0.2547		mg/l	0.00100	0.00012	1	01/07/16 09:20	01/07/16 13:51	EPA 3005A	1,6020A	KL

ALPHA
ANALYTICAL

Project Name:	WURTSBORO MINE		Lab Number:	L1600382
Project Number:	WURTSBORO MINE		Report Date:	01/11/16

SAMPLE RESULTS

Lab ID:	L1600382-05	Date Collected:	01/05/16 13:30
Client ID:	21 LITERS 3/8" LIMESTONE	Date Received:	01/06/16
Sample Location:	WURTSBORO, NY	Field Prep:	Not Specified
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Prep Method	Analytical Method	Analyst
Total Metals - Westborough Lab											
Lead, Total	0.3557		mg/l	0.00100	0.00012	1	01/07/16 09:20	01/07/16 14:14	EPA 3005A	1,6020A	KL

ALPHA
ANALYTICAL

| Project Name: | WURTSBORO MINE | | Lab Number: | L1600382 |
| Project Number: | WURTSBORO MINE | | Report Date: | 01/11/16 |

SAMPLE RESULTS

Lab ID:	L1600382-06	Date Collected:	01/05/16 16:05
Client ID:	39 LITERS 3/8" LIMESTONE	Date Received:	01/06/16
Sample Location:	WURTSBORO, NY	Field Prep:	Not Specified
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Prep Method	Analytical Method	Analyst
Total Metals - Westborough Lab											
Lead, Total	0.3835		mg/l	0.00100	0.00012	1	01/07/16 09:20	01/07/16 14:25	EPA 3005A	1,6020A	KL

| Project Name: | WURTSBORO MINE | | Lab Number: | L1600382 |
| Project Number: | WURTSBORO MINE | | Report Date: | 01/11/16 |

SAMPLE RESULTS

Lab ID:	L1600382-07	Date Collected:	01/06/16 10:00
Client ID:	70 LITERS 3/8" LIMESTONE	Date Received:	01/06/16
Sample Location:	WURTSBORO, NY	Field Prep:	Not Specified
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Prep Method	Analytical Method	Analyst
Total Metals - Westborough Lab											
Lead, Total	0.4104		mg/l	0.00100	0.00012	1	01/07/16 09:20	01/07/16 14:29	EPA 3005A	1,6020A	KL

ALPHA

Project Name:	WURTSBORO MINE	Lab Number:	L1600382
Project Number:	WURTSBORO MINE	Report Date:	01/11/16

SAMPLE RESULTS

Lab ID:	L1600382-08	Date Collected:	01/06/16 10:00
Client ID:	70 LITERS 3/8" LIMESTONE	Date Received:	01/06/16
Sample Location:	WURTSBORO, NY	Field Prep:	Not Specified
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Prep Method	Analytical Method	Analyst
Dissolved Metals - Westborough Lab											
Lead, Dissolved	0.3043		mg/l	0.00100	0.00012	1	01/07/16 12:05	01/07/16 16:10	EPA 3005A	1,6020A	KL

ALPHA

Project Name: WURTSBORO MINE

Lab Number: L1600382

Project Number: WURTSBORO MINE

Report Date: 01/11/16

Method Blank Analysis
Batch Quality Control

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
Total Metals - Westborough Lab for sample(s): 01,03-07 Batch: WG855399-1										
Lead, Total	ND		mg/l	0.00100	0.00012	1	01/07/16 09:20	01/07/16 13:40	1,6020A	KL

Prep Information

Digestion Method: EPA 3005A

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
Dissolved Metals - Westborough Lab for sample(s): 02,08 Batch: WG855459-1										
Lead, Dissolved	ND		mg/l	0.00100	0.00012	1	01/07/16 12:05	01/07/16 15:33	1,6020A	KL

Prep Information

Digestion Method: EPA 3005A

ΔLPHA
ANALYTICAL

Lab Control Sample Analysis
Batch Quality Control

Project Name: WURTSBORO MINE

Project Number: WURTSBORO MINE

Lab Number: L1600382

Report Date: 01/11/16

Parameter	LCS %Recovery	Qual	LCSD %Recovery	Qual	%Recovery Limits	RPD	Qual	RPD Limits
Total Metals - Westborough Lab Associated sample(s): 01,03-07 Batch: WG855399-2								
Lead, Total	105		-		80-120	-		
Dissolved Metals - Westborough Lab Associated sample(s): 02,08 Batch: WG855459-2								
Lead, Dissolved	107		-		80-120	-		

ALPHA
ANALYTICAL

Matrix Spike Analysis
Batch Quality Control

Project Name:	WURTSBORO MINE	**Lab Number:**	L1600382
Project Number:	WURTSBORO MINE	**Report Date:**	01/11/16

Parameter	Native Sample	MS Added	MS Found	MS %Recovery	Qual	MSD Found	MSD %Recovery	Qual	Recovery Limits	RPD	Qual	RPD Limits
Total Metals - Westborough Lab Associated sample(s): 01,03-07 QC Batch ID: WG855399-4 QC Sample: L1600382-04 Client ID: 8 LITERS 3/8" LIMESTONE												
Lead, Total	0.2547	0.51	0.8126	109		-	-		75-125	-		20
Dissolved Metals - Westborough Lab Associated sample(s): 02,08 QC Batch ID: WG855459-4 QC Sample: L1600382-02 Client ID: UNTREATED 2												
Lead, Dissolved	0.4754	0.51	1.056	114		-	-		75-125	-		20

ALPHA

Lab Duplicate Analysis
Batch Quality Control

Project Name:	WURTSBORO MINE	**Lab Number:**	L1600382
Project Number:	WURTSBORO MINE	**Report Date:**	01/11/16

Parameter	Native Sample	Duplicate Sample	Units	RPD	Qual	RPD Limits
Total Metals - Westborough Lab Associated sample(s): 01,03-07 QC Batch ID: WG855399-3 QC Sample: L1600382-04 Client ID: 8 LITERS 3/8" LIMESTONE						
Lead, Total	0.2547	0.2600	mg/l	2		20
Dissolved Metals - Westborough Lab Associated sample(s): 02,08 QC Batch ID: WG855459-3 QC Sample: L1600382-02 Client ID: UNTREATED 2						
Lead, Dissolved	0.4754	0.4797	mg/l	1		20

ALPHA

Project Name: WURTSBORO MINE

Project Number: WURTSBORO MINE

Lab Number: L1600382

Report Date: 01/11/16

Sample Receipt and Container Information

Were project specific reporting limits specified? YES

Cooler Information Custody Seal

Cooler

A   Absent

Container Information

Container ID	Container Type	Cooler	pH	Temp deg C	Pres	Seal	Analysis(*)
L1600382-01A	Plastic 250ml HNO3 preserved	A	<2	1.9	Y	Absent	PB-6020T(180)
L1600382-02A	Plastic 250ml unpreserved	A	7	1.9	Y	Absent	-
L1600382-02X	Plastic 120ml HNO3 preserved spl	A	<2	1.9	Y	Absent	PB-6020S(180)
L1600382-03A	Plastic 250ml HNO3 preserved	A	<2	1.9	Y	Absent	PB-6020T(180)
L1600382-04A	Plastic 250ml HNO3 preserved	A	<2	1.9	Y	Absent	PB-6020T(180)
L1600382-05A	Plastic 250ml HNO3 preserved	A	<2	1.9	Y	Absent	PB-6020T(180)
L1600382-06A	Plastic 250ml HNO3 preserved	A	<2	1.9	Y	Absent	PB-6020T(180)
L1600382-07A	Plastic 500ml HNO3 preserved	A	<2	1.9	Y	Absent	PB-6020T(180)
L1600382-08A	Plastic 250ml unpreserved	A	7	1.9	Y	Absent	-
L1600382-08X	Plastic 120ml HNO3 preserved spl	A	<2	1.9	Y	Absent	PB-6020S(180)

*Values in parentheses indicate holding time in days

ALPHA

GLOSSARY

Acronyms

EDL	- Estimated Detection Limit: This value represents the level to which target analyte concentrations are reported as estimated values, when those target analyte concentrations are quantified below the reporting limit (RL). The EDL includes any adjustments from dilutions, concentrations or moisture content, where applicable. The use of EDLs is specific to the analysis of PAHs using Solid-Phase Microextraction (SPME).
EPA	- Environmental Protection Agency.
LCS	- Laboratory Control Sample: A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes.
LCSD	- Laboratory Control Sample Duplicate: Refer to LCS.
LFB	- Laboratory Fortified Blank: A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes.
MDL	- Method Detection Limit: This value represents the level to which target analyte concentrations are reported as estimated values, when those target analyte concentrations are quantified below the reporting limit (RL). The MDL includes any adjustments from dilutions, concentrations or moisture content, where applicable.
MS	- Matrix Spike Sample: A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.
MSD	- Matrix Spike Sample Duplicate: Refer to MS.
NA	- Not Applicable.
NC	- Not Calculated: Term is utilized when one or more of the results utilized in the calculation are non-detect at the parameter's reporting unit.
NI	- Not Ignitable.
NP	- Non-Plastic: Term is utilized for the analysis of Atterberg Limits in soil.
RL	- Reporting Limit: The value at which an instrument can accurately measure an analyte at a specific concentration. The RL includes any adjustments from dilutions, concentrations or moisture content, where applicable.
RPD	- Relative Percent Difference: The results from matrix and/or matrix spike duplicates are primarily designed to assess the precision of analytical results in a given matrix and are expressed as relative percent difference (RPD). Values which are less than five times the reporting limit for any individual parameter are evaluated by utilizing the absolute difference between the values; although the RPD value will be provided in the report.
SRM	- Standard Reference Material: A reference sample of a known or certified value that is of the same or similar matrix as the associated field samples.
STLP	- Semi-dynamic Tank Leaching Procedure per EPA Method 1315.
TIC	- Tentatively Identified Compound: A compound that has been identified to be present and is not part of the target compound list (TCL) for the method and/or program. All TICs are qualitatively identified and reported as estimated concentrations.

Footnotes

1   - The reference for this analyte should be considered modified since this analyte is absent from the target analyte list of the original method.

Terms

Total: With respect to Organic analyses, a 'Total' result is defined as the summation of results for individual isomers or Aroclors. If a 'Total' result is requested, the results of its individual components will also be reported. This is applicable to 'Total' results for methods 8260, 8081 and 8082.

Analytical Method: Both the document from which the method originates and the analytical reference method. (Example: EPA 8260B is shown as 1,8260B.) The codes for the reference method documents are provided in the References section of the Addendum.

Data Qualifiers

A   - Spectra identified as "Aldol Condensation Product".

B   - The analyte was detected above the reporting limit in the associated method blank. Flag only applies to associated field samples that have detectable concentrations of the analyte at less than ten times (10x) the concentration found in the blank. For MCP-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte at less than ten times (10x) the concentration found in the blank. For DOD-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte at less than ten times (10x) the concentration found in the blank AND the analyte was detected above one-half the reporting limit (or above the reporting limit for common lab contaminants) in the associated method blank. For NJ-Air-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte above the reporting limit. For NJ-related projects (excluding Air), flag only applies to associated field samples that have detectable concentrations of the analyte, which was detected above the reporting limit in the associated method blank or above five times the reporting limit for common lab contaminants (Phthalates, Acetone, Methylene Chloride, 2-Butanone).

Report Format: DU Report with 'J' Qualifiers

ALPHA

| Project Name: | WURTSBORO MINE | Lab Number: | L1600382 |
| Project Number: | WURTSBORO MINE | Report Date: | 01/11/16 |

Data Qualifiers

C — Co-elution: The target analyte co-elutes with a known lab standard (i.e. surrogate, internal standards, etc.) for co-extracted analyses.

D — Concentration of analyte was quantified from diluted analysis. Flag only applies to field samples that have detectable concentrations of the analyte.

E — Concentration of analyte exceeds the range of the calibration curve and/or linear range of the instrument.

G — The concentration may be biased high due to matrix interferences (i.e. co-elution) with non-target compound(s). The result should be considered estimated.

H — The analysis of pH was performed beyond the regulatory-required holding time of 15 minutes from the time of sample collection.

I — The lower value for the two columns has been reported due to obvious interference.

M — Reporting Limit (RL) exceeds the MCP CAM Reporting Limit for this analyte.

NJ — Presumptive evidence of compound. This represents an estimated concentration for Tentatively Identified Compounds (TICs), where the identification is based on a mass spectral library search.

P — The RPD between the results for the two columns exceeds the method-specified criteria.

Q — The quality control sample exceeds the associated acceptance criteria. For DOD-related projects, LCS and/or Continuing Calibration Standard exceedences are also qualified on all associated sample results. Note: This flag is not applicable for matrix spike recoveries when the sample concentration is greater than 4x the spike added or for batch duplicate RPD when the sample concentrations are less than 5x the RL. (Metals only.)

R — Analytical results are from sample re-analysis.

RE — Analytical results are from sample re-extraction.

S — Analytical results are from modified screening analysis.

J — Estimated value. The Target analyte concentration is below the quantitation limit (RL), but above the Method Detection Limit (MDL) or Estimated Detection Limit (EDL) for SPME-related analyses. This represents an estimated concentration for Tentatively Identified Compounds (TICs).

ND — Not detected at the method detection limit (MDL) for the sample, or estimated detection limit (EDL) for SPME-related analyses.

Report Format: DU Report with 'J' Qualifiers

Project Name:	WURTSBORO MINE	Lab Number:	L1600382
Project Number:	WURTSBORO MINE	Report Date:	01/11/16

REFERENCES

1   Test Methods for Evaluating Solid Waste: Physical/Chemical Methods. EPA SW-846. Third Edition. Updates I - IV, 2007.

LIMITATION OF LIABILITIES

Alpha Analytical performs services with reasonable care and diligence normal to the analytical testing laboratory industry. In the event of an error, the sole and exclusive responsibility of Alpha Analytical shall be to re-perform the work at it's own expense. In no event shall Alpha Analytical be held liable for any incidental, consequential or special damages, including but not limited to, damages in any way connected with the use of, interpretation of, information or analysis provided by Alpha Analytical.

We strongly urge our clients to comply with EPA protocol regarding sample volume, preservation, cooling, containers, sampling procedures, holding time and splitting of samples in the field.

Alpha Analytical, Inc.
Facility: **Company-wide**
Department: **Quality Assurance**
Title: **Certificate/Approval Program Summary**

Certification Information

The following analytes are not included in our **Primary NELAP Scope of Accreditation:**

Westborough Facility
EPA 524.2: 1,2-Dibromo-3-chloropropane, 1,2-Dibromoethane, m/p-xylene, o-xylene
EPA 624: 2-Butanone (MEK), 1,4-Dioxane, tert-Amylmethyl Ether, tert-Butyl Alcohol, m/p-xylene, o-xylene
EPA 625: Aniline, Benzoic Acid, Benzyl Alcohol, 4-Chloroaniline, 3-Methylphenol, 4-Methylphenol.
EPA 1010A: <u>NPW:</u> Ignitability
EPA 6010C: <u>NPW:</u> Strontium; <u>SCM:</u> Strontium
EPA 8151A: <u>NPW:</u> 2,4-DB, Dicamba, Dichloroprop, MCPA, MCPP; <u>SCM:</u> 2,4-DB, Dichloroprop, MCPA, MCPP
EPA 8260C: <u>NPW:</u> 1,2,4,5-Tetramethylbenzene; 4-Ethyltoluene, Azobenzene, Isopropanol; <u>SCM:</u> Iodomethane (methyl iodide), Methyl methacrylate (soil); 1,2,4,5-Tetramethylbenzene; 4-Ethyltoluene.
EPA 8270D: <u>NPW:</u> Pentachloronitrobenzene, 1-Methylnaphthalene, Dimethylnaphthalene,1,4-Diphenylhydrazine; <u>SCM:</u> Pentachloronitrobenzene, 1-Methylnaphthalene, Dimethylnaphthalene,1,4-Diphenylhydrazine.
EPA 9010: <u>NPW:</u> Amenable Cyanide Distillation, Total Cyanide Distillation
EPA 9038: <u>NPW:</u> Sulfate
EPA 9050A: <u>NPW:</u> Specific Conductance
EPA 9056: <u>NPW:</u> Chloride, Nitrate, Sulfate
EPA 9065: <u>NPW:</u> Phenols
EPA 9251: <u>NPW:</u> Chloride
SM3500: <u>NPW:</u> Ferrous Iron
SM4500: <u>NPW:</u> Amenable Cyanide, Dissolved Oxygen; <u>SCM:</u> Total Phosphorus, TKN, NO2, NO3.
SM5310C: <u>DW:</u> Dissolved Organic Carbon

Mansfield Facility
EPA 8270D: <u>NPW:</u> Biphenyl; <u>SCM:</u> Biphenyl
EPA 2540D: TSS
EPA TO-15: Halothane, 2,4,4-Trimethyl-2-pentene, 2,4,4-Trimethyl-1-pentene, Thiophene, 2-Methylthiophene, 3-Methylthiophene, 2-Ethylthiophene, 1,2,3-Trimethylbenzene, Indan, Indene, 1,2,4,5-Tetramethylbenzene, Benzothiophene, 1-Methylnaphthalene.

The following analytes are included in our **Massachusetts DEP Scope of Accreditation, Westborough Facility:**

Drinking Water
EPA 200.8: Sb,As,Ba,Be,Cd,Cr,Cu,Pb,Ni,Se,Tl; **EPA 200.7:** Ba,Be,Ca,Cd,Cr,Cu,Na; **EPA 245.1:** Mercury;
EPA 300.0: Nitrate-N, Fluoride, Sulfate; **EPA 353.2:** Nitrate-N, Nitrite-N; **SM4500NO3-F:** Nitrate-N, Nitrite-N; **SM4500F-C, SM4500CN-CE, EPA 180.1, SM2130B, SM4500Cl-D, SM2320B, SM2540C, SM4500H-B**
EPA 332: Perchlorate.
Microbiology: SM9215B; SM9223-P/A, SM9223B-Colilert-QT, Enterolert-QT.

Non-Potable Water
EPA 200.8: Al,Sb,As,Be,Cd,Cr,Cu,Pb,Mn,Ni,Se,Ag,Tl,Zn;
EPA 200.7: Al,Sb,As,Be,Cd,Ca,Cr,Co,Cu,Fe,Pb,Mg,Mn,Mo,Ni,K,Se,Ag,Na,Sr,Ti,Tl,V,Zn;
EPA 245.1, SM4500H,B, EPA 120.1, SM2510B, SM2540C, SM2340B, SM2320B, SM4500CL-E, SM4500F-BC, SM426C, SM4500NH3-BH, EPA 350.1: Ammonia-N, **LACHAT 10-107-06-1-B:** Ammonia-N, **SM4500NO3-F,**
EPA 353.2: Nitrate-N, **SM4500NH3-BC-NES, EPA 351.1, SM4500P-E, SM4500P-B, E, SM5220D, EPA 410.4, SM5210B, SM5310C, SM4500CL-D, EPA 1664, SM14 510AC, EPA 420.1, SM4500-CN-CE, SM2540D.**
EPA 624: Volatile Halocarbons & Aromatics,
EPA 608: Chlordane, Toxaphene, Aldrin, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, Dieldrin, DDD, DDE, DDT, Endosulfan I, Endosulfan II, Endosulfan sulfate, Endrin, Endrin Aldehyde, Heptachlor, Heptachlor Epoxide, PCBs
EPA 625: SVOC (Acid/Base/Neutral Extractables), **EPA 600/4-81-045:** PCB-Oil.
Microbiology: SM9223B-Colilert-QT; Enterolert-QT, SM9222D-MF.

For a complete listing of analytes and methods, please contact your Alpha Project Manager.

Document Type: Form

Pre-Qualtrax Document ID: 08-113

NEW JERSEY CHAIN OF CUSTODY

ALPHA

Westborough, MA 01581
8 Walkup Dr.
TEL: 508-898-9220
FAX: 508-898-9193

Mansfield, MA 02048
320 Forbes Blvd
TEL: 508-822-9300
FAX: 508-822-3288

Service Centers
Mahwah, NJ 07430: 35 Whitney Rd, Suite 5
Albany, NY 12205: 14 Walker Way
Tonawanda, NY 14150: 275 Cooper Ave, Suite 105

Page 1 of 1

Date Rec'd In Lab: 1/6/16

ALPHA Job #: C16C0382

Project Information

Project Name: WURTSBORO MINE
Project Location: WURTSBORO NY
Project #:
(Use Project name as Project #) ☒
Project Manager: ROBERT KUNZEL
ALPHAQuote #:

Turn-Around Time
Standard ☐
Rush (only if pre approved) ☒
Due Date: 1/11/16
# of Days:

These samples have been previously analyzed by Alpha ☐

Client Information
Client: GROUND/WATER TST
Address: 627 MT HOPE RD
WHARTON NJ 07885
Phone: 973 983 0901
Fax: 973 983 0903
Email: RKUNZEL CGWTLLC.COM

Deliverables
☐ NJ Full / Reduced
☐ EQuIS (1 File) ☐ EQuIS (4 File)
☐ Other

Regulatory Requirement
☐ SRS Residential/Non Residential
☐ SRS Impact to Groundwater
☐ NJ Ground Water Quality Standards
☐ NJ IGW SPLP Leachate Criteria
☒ Other EPA

Billing Information
☒ Same as Client Info
PO #: 09 RGK

Site Information
Is this site impacted by Petroleum? Yes ☐ No
Petroleum Product:

For EPH, selection is REQUIRED:
☐ Category 1
☐ Category 2

For VOC, selection is REQUIRED:
☐ 1,4-Dioxane
☐ 8011

Other project specific requirements/comments:

Please specify Metals or TAL.

Sample Filtration
☐ Done
☐ Lab to do
Preservation
☐ Lab to do

(Please Specify below)

ALPHA Lab ID (Lab Use Only)	Sample ID	Collection Date	Collection Time	Sample Matrix	Sampler's Initials	TOTAL LEAD	DISSOLVED LEAD					Sample Specific Comments
0382-01	UNTREATED Z	1/5/16	10:00	W	RGK	X						
02	UNTREATED Z	1/5/16	10:00	W	RGK		X					
03	3 LITERS 3/8" LIMESTONE	1/5/16	10:35	W	RGK	X						
04	8 '' ''	1/5/16	11:25	W	RGK	X						
05	21 '' ''	1/5/16	13:30	W	RGK	X						
06	39 '' ''	1/5/16	16:05	W	RGK	X						
07	70 '' ''	1/6/16	10:00	W	RGK	X						
08	70 '' ''	1/6/16	10:00	W	RGK		X					

Preservative Code:
A = None
B = HCl
C = HNO₃
D = H₂SO₄
E = NaOH
F = MeOH
G = NaHSO₄
H = Na₂S₂O₃
K/E = Zn Ac/NaOH
O = Other

Container Code:
P = Plastic
A = Amber Glass
V = Vial
G = Glass
B = Bacteria Cup
C = Cube
O = Other
E = Encore
D = BOD Bottle

Westboro: Certification No: MA935
Mansfield: Certification No: MA015

Container Type: PLASTIC
Preservative:

Relinquished By:	Date/Time	Received By:	Date/Time
[signature]	1/6/16 11:00	Joan Hooghkirk ACT	1/6/16 1730
KUNZEL		Tom Tobin	1-6-16 1830
Joan Hooghkirk	1/6/16 1830		
Tom Tobin	1-6-16 2140	Al Williams	1-6-16 2140
Al Williams	1/6/16 2340	[signature]	1/6/16 2340

Form No: 01-14 HC (rev. 30-Sept-2013)